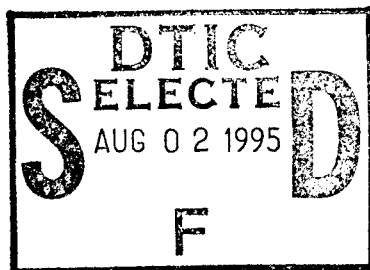
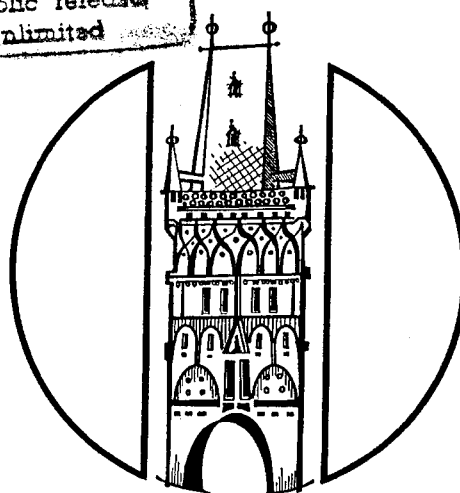


# Final Program and Abstracts



## Seventh International Symposium on Nondestructive Characterization of Materials

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Czech Technical University  
Prague, Czech Republic  
June 19-22, 1995

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# **SEVENTH INTERNATIONAL SYMPOSIUM ON NONDESTRUCTIVE CHARACTERIZATION OF MATERIALS™**

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## **Co-Chairs**

Anthony L. Bartos  
Computer Sciences Corporation  
USA

Clayton O. Ruud  
The Pennsylvania State University  
USA

Robert E. Green, Jr.  
The Johns Hopkins University  
USA

Miroslava Vrbova  
Czech Technical University, Prague  
Czech Republic

Jaroslav Obrázek  
President, Czech NDT Society  
Czech Republic

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## **Supported in part by:**

The American Society for Nondestructive Testing, Inc.; the National Institute of Standards & Technology; and the National Science Foundation

## **Acknowledge:**

We wish to additionally thank the US Air Force European Office of Aerospace Research and Development, and the U.S. Army European Research Office for its contribution to the success of the Symposium

## International Organizing Committee

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### **Presentations**

The goal of this symposium is to include as many oral presentations as possible. There will be parallel sessions on Monday (AM & PM), Tuesday & Wednesday (AM), and Thursday (AM & PM).

### **Featured Sessions**

Magnetic Techniques  
Non-Metallic Materials  
Stress Measurement  
X-ray Applications  
Optical Techniques Including Laser  
Ultrasound  
Thermal Techniques  
Posters  
Acoustic Emission and Internal Friction  
Composite Materials  
Process Control  
Particle Technology  
Ultrasonics Applications  
Basic Ultrasonics

### **Audio/Visual**

You are scheduled to meet with your session chairman and audio/visual coordinator approximately (10) minutes before your session. Please bring your slides and/or other visual aides to this meeting. Your oral presentation is scheduled for the duration of (20) minutes allowing time for a question and answer period.

### **Posters**

If you did not bring a picture for display with your poster board, please stop by the registration desk to have one taken.

### **Guidelines for Authors**

You must be a registered author of the accepted abstract to present the paper. Your paper cannot be presented by someone else unless they are an author whose name is listed on the abstract.

### **Proceedings**

The proceedings of this symposium will be published as "Nondestructive Characterization of Materials VII" by Trans Tech Publications Ltd (Email [wohlbier@transtech.ch](mailto:wohlbier@transtech.ch))

### **Manuscript Deadline**

If your final paper is not submitted during the symposium, it must be postmarked by August 1, 1995 to the symposium coordinator. If you are a paid attendee, and your paper is received before the deadline, you will be guaranteed a free copy of the proceedings.

### **Message Board**

Next to registration desk

### **Mail List**

A list of attendees will be mailed to everyone after the symposium

**Tours**

Golden City Tours will have tour packages available at the registration area.

**Social Function**

*Monday, June 19*

Opening Reception

Bethlehem Chapel

7:30-10 PM

Address: Bethlehem Square

Prague 1 - Old Town

*Wednesday, June 21*

Afternoon Tours

*Thursday, June 22*

Farewell Banquet,

Smichov Brewery Restaurant

Staropramen

7:30-10 PM

Address: Nadrazni 84

Prague 5-Smichov

**Proceedings**

All papers (invited, contributed and poster presentations) will be included in the Proceedings. Detailed information on how to prepare manuscripts and posters for the Proceedings have been provided directly to the first author.

**Symposium Coordinator**

Debbie Harris

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**Proceedings**

The Sixth International Symposium on Nondestructive Characterization of Materials was held in Oahu, Hawaii on June 7-11, 1993. The proceedings are available from the symposium coordinator.

**NOTE: Highlighted papers are "INVITED"**

## I. MAGNETIC TECHNIQUES

**Monday, June 19                      Session (A) AM**

*Chairs: M. Kroning, Fraunhofer-Inst. for NDT, Germany & J. Štájer, Advanced Technology Group, Czech Republic*

**8:30 Early Recognition of H-Induced Stress Corrosion Cracking with Micromagnetic Testing Methods—M. Lang & I. Altpeter, Fraunhofer-Institute for Nondestructive Testing Universität, Germany**

**8:50 The Development of Nondestructive Evaluation (NDE) for Monitoring the Embrittlement in Nuclear Reactor Pressure Vessels—M. Blaszkiewicz, Westinghouse Science and Technology Center, USA**

**9:10 Evaluation of Digitized Signals From Defectoscopic Checking of Steel Ropes—O. Lesnák, Research Mining Institute, Czech Republic**

**9:30 Detection of Variations in Heat Treatment and Conductivity in Metals Using Surface Magnetic Field Measurement Technique—D. Mirshekar-Syahkal & R. Mostafavi, University of Essex, United Kingdom**

**9:50 Ferromagnetic Surface Layers Testing with Depth Resolution Using a priori Knowledge—V. Vengrinovich & S. Zolotarev, Belarussian Academy of Sciences, Belarussia**

**10:10 BREAK**

**10:30 Barkhausen Analysis of the Effect of Strain and Heat Treatment on Epsilon-Martensite—I. Mészáros & M. Káldor, Technical University of Budapest, Hungary**

**10:50 Peculiarities of Connection Between Mechanical Properties and Residual Magnetization of Articles of Different Size—S. Sandomirskii, Belarussian Academy of Sciences, Belarussia**

**11:10 Non-Destructive Measurements of Grain size in Steel Plate by Using Magnetic Coercive Force—M. Yoshino & H. Tanabe, NKK Corp.; T. Sakamoto, Sumitomo Metal Ind. Ltd.; N. Suzuki, Kobe Steel Ltd.; & Y. Yaji, Nippon Steel Corp., Japan**

**11:30 Detection of the Tendency of Chilling in Series Manufactured Cast Iron Components Using Micromagnetic Testing Procedures—M. Kröning & I. Altpeter, Fraunhofer-Institute for Nondestructive Testing, & U. Laub, Q NET GmbH, Germany**

**11:50 Nondestructive Determination of Elastic into the Microplastic State Transition—L. Keller & P. Stanek, TSI System s.r.o., Military Technical Institute of Protection, Czech Republic**

**12:10 LUNCH**

## II. NON-METALLIC MATERIALS

**Monday, June 19                      Session (A) PM**

*Chairs: R. Zoughi, Colorado State University & G. Hagnauer, Army Research Lab., USA*

**1:30 Overview of Microwave NDE Applied to Thick Composites—R. Zoughi & S. Ganchev, Colorado State University & G. Carneau, NTIAC-TRI, USA**

**1:50 Characterization of Green Ceramics by Microwaves and Ultrasound—M. Kröning, R. Schneider, & U. Netzelmann, Fraunhofer-Institut für Zerstörungsfreie Prüfverfahren, Germany**

**2:10 Non-destructive Moisture Measurement Using Microwaves—F. Thompson, Manchester Metropolitan Univ., United Kingdom**

**2:30 Defect Characterization by a Microwave Testing System at 30 GHz compared with Results of other NDE-Methods—L. Diener, D. Wu, W. Rippel, R. Steegmüller, A. Schmid, & G. Busse, Institut für Kunststoffprüfung und Kunststoffkunde, Germany**

**2:50 Models for Microwave Nondestructive Testing of Materials—N. Ida, The University of Akron, USA**

**3:10 BREAK**

**3:30 Acoustics of Wood—V. Bucur, Institut National de la Recherche Agronomique & Université "Henri Poincaré," Nancy, Laboratoire d'Etudes et Recherches sur le Bois, France**

**3:50 Nondestructive Evaluation of Logs for Structural Product Quality—R. Ross, K. McDonald, K. Schad, & D. Green, USDA Forest Products Laboratory, USA**

**4:10 Vibrations of Piano Soundboards - Real Soundboard without Ribs in Comparison with its FEM Model—J. Skala & A. Raffaj, Petrof Piano Factory, Czech Republic**

**4:30 Durability Assessment of Polymer Matrix Composite Materials—G. Hagnauer, A. Gutierrez, & J. Kleinmeyer, US Army Research Laboratory, USA**

**4:50 ADJOURN**

### **III. STRESS MEASUREMENT**

**Monday, June 19 Session (B) AM**

*Chairs: K. Kozaczek, Oak Ridge National Lab., USA & E. Schneider, Fraunhofer-Inst. for NDT, Germany*

**8:30 Measurement of Dislocation Density by Residual Electrical Resistivity—M. Kocer, F. Sachslehner, M. Müller, E. Schafner, & M. Zehetbauer, Universität Wien, Austria**

**8:50 Anisotropy of Young's Modulus and Technological Properties—R. Fiedler, TU Brno; & J. Zeman, Military Technical Institute, Czech Republic**

**9:10 Residual Stress Depth Profiles of Ausrolled 9310 Gear Steel—C. Paliani & R. Queeney, The Pennsylvania State University; & K. Kozaczek, Oak Ridge National Laboratory, USA**

**9:30 Neutron Diffraction Residual Stress Measurement at NIST—H. Prask, National Institute of Standards and Technology; & P. Brand, University of Maryland, USA**

**9:50 On the Calibration of Magnetic and Ultrasonic Methods of Residual Stress Measurements in Cold Rolled Iron-Disks by Neutron Diffraction Technique—G. Bokuchava & Y. Taran, Frank Laboratory of Neutron Physics, Russia; K. Herold, Fraunhofer-Einrichtung IUW Chemnitz; & E. Schneider, J. Schreiber, & W. Theiner, Fraunhofer-Institute for Nondestructive Testing, Germany**

**10:10 BREAK**

**10:30 Localized Stress Measurement of Aluminum Alloy with an Acoustic Microscope**—M. Okade, Aisin Seiki Co. Ltd.; & K. Kawashima, Nagoya Institute of Technology, Japan

**10:50 Ultrasonic Evaluation of Stress States in Rails**—E. Schneider, R. Herzer, D. Bruche, & M. Kröning, Fraunhofer Institute for Nondestructive Testing, Germany

**11:10 Acoustoelastic Determination of Stresses in Steel Using Rayleigh Ultrasonic Waves**—T. Berruti & M. Gala, Politecnico di Torino, Italy

**11:30 The Eddy Current Technique for Determining Residual Stresses in Steels**—M. Blaszkiewicz & L. Albertin, Westinghouse Science and Technology Center, USA

**11:50 Another Approach to Acoustoelasticity**—E. Krasicka, National Institute of Standards and Technology, USA

**12:10 LUNCH BREAK**

#### **IV. X-RAY APPLICATIONS**

**Monday, June 19 Session (B) PM**

*Chairs: C. Landron, Centre De Recherches Sur, France & Z. Zavadil, Advanced Technology Group, Czech Republic*

**1:30 Recent Developments in Non-Destructive Characterization of Aerosols By Synchrotron Radiation**—C. Landron, Centre de Recherches sur la Physique des Hautes Températures, France

**1:50 Determination of Single Crystal Orientation From Oscillatory Bragg Peak Position**—D. Dragoi, University of Denver; & K. Kozaczek & T. Watkins, Oak Ridge National Laboratory, USA

**2:10 Nondestructive Measurement of Grain Size in Steel Plate by Using X-ray Diffraction**—F. Ichikawa & M. Okuno, Kawasaki Steel Corp.; & T. Tanaka & M. Okamoto, Nippon Steel Corp., Japan

**2:30 Hydriding Characteristics of V-0.5 at .%C Alloy**—D. Chandra, A. Sharma, & W. Cathey, University of Nevada; F. Lynch, Hydrogen Consultants Inc.; & R. Bowman, Jr., Aerojet Electronic Systems Division, USA

**2:50 X-ray Diffraction Characterization of Thin Polycrystalline Films**—K. Kozaczek & T. Watkins, Oak Ridge National Laboratory; G. Book & W. Carter, Georgia Institute of Technology; & A. Hunt, CCVD, Inc. USA

**3:10 BREAK**

**3:30 Principle of Practice of Modified Proportional Factor Method in XRF Analysis**—C. Yuanpan, China National Nonferrous Metals Industry Corporation, China

**3:50 X-ray Backscatter Tomography: NDT Potential and Limitations**—C. Poranski & Y. Ham, Naval Research Lab.; & E. Greenawald, Geo-Centers, Inc., USA

**4:10 A X-ray Sensitive CCD Camera System and its Application to the X-ray Diffractometric Investigation of Area Selective Semiconductor Epitaxy**—F. Frandich & R. Koehler, MPG-Arbeitsgruppe, Germany

**4:30 ADJOURN**



## **V. OPTICAL TECHNIQUES INCLUDING LASER ULTRASOUND**

**Tuesday, June 20                      Session (A) AM**

*Chairs: R. Dewhurst, WMIST, UK & K. Škvor, Czech Technical University, Czech Republic*

**8:30    Determination of the Elastic Behaviour of Carbon-Reinforced Carbon Materials by Using Laser-Ultrasonics and Theoretical Modelling—M. Spies, B. Haberer, M. Paul, & W. Arnold, Fraunhofer Institute for Nondestructive Testing, Germany**

**8:50    Elastic Moduli Measurements of SiC Reinforced Alumina Ceramics at High Temperatures Using Laser-Ultrasonics—A. Moreau, National Research Council of Canada; & F. Taheri, Technical University of Nova Scotia, Canada**

**9:10    Characterization of Creep Damage by Absorption Measurements Using Laser Ultrasound—P. Kalyanasundaram, Indira Gandhi Centre for Atomic Research, India; & J. Reszat, & M. Paul, Fraunhofer Institut für zerstörungsfreie Prüfverfahren, Germany**

**9:30    Noncontact Alternative to Laser Detection of Ultrasonic Signals—J. Wagner, D. Oursler, & T. Murray, The Johns Hopkins University, USA**

**9:50    Nickel-base Superalloys Characterized by SLAM After Long Term Heating—V. Luprano & G. Montagna, Pastis-CNRS, Italy**

**10:10    BREAK**

**10:30    LMM-1 Laser Microanalyser of Materials—A. Kotyuk, M. Ulanovsky, V. Arbekov, & V. Kuznetsov, The All-Russian Research Institute for Optical and Physical Measurements, Russia**

**10:50    Microhardness and Raman Spectroscopy for Characterization of Fullerite Single Crystals—M. Haluska, M. Zehetbauer, & H. Kuzmany, Institut für Festkörperphysik, Universität Wien, Austria**

**11:10    Very Near Field Optics, A Frontier Technology—J. Goodell, Westinghouse, USA**

**11:30    Delayed Cracking in Automotive Windshields—S. Gulati, H. Hagy, & J. Bayne, Corning Incorporated, USA**

**11:50    High-Bandwidth, Self-Compensating, Laser-Based Ultrasound Detector Using Photo-Induced EMF in GaAs—P. Mitchell, S. McCahon, M. Klein, T. O'Meara, G. Dunning, & D. Pepper, Hughes Research Laboratories, USA**

**12:10    LUNCH BREAK**

## **VI. THERMAL TECHNIQUES**

**Tuesday, June 20                      Session (B) AM**

*Chairs: K. Kawashima, Nagoya Inst. of Tech., Japan & G. Busse, Institut für Kunststoffprüfung und Kunststoffkunde, Germany*

**8:30    Lock-in Vibrothermography Applied for NDT of Polymer Materials—J. Rantala, D. Wu & G. Busse, Institut für Kunststoffprüfung und Kunststoffkunde, Germany**

**8:50    Photothermal Investigation of Silicon Wafers with Diamond-like coating—J. Bodzenta, J. Mazur, & R. Bukowski, Silesian Technical University, Poland**

**9:10 High Resolution Photothermal Imaging of Metal Matrix Composite Interface**—F. Chen, I-Lan Institute of Agriculture and Technology, Taiwan, R.O.C.; & U. Netzelmann, M. Disque & M. Kröning, Fraunhofer Institute for Nondestructive Testing, Germany

**9:30 Nondestructive Testing of Lacquer Coatings**—B. Bendjus, B. Koehler, & Th. Vetterlein, EADQ, Germany

**9:50 Structural and Morphological Characterization of Particulate Ceramic Materials by Infrared Spectroscopy**—M. Ocaña & C. Serna, Instituto de Ciencia de Materiales de Madrid, Spain

**10:10 BREAK**

**10:30 Evolved Heat as a Fatigue Characterizing Parameter**—N. Rajic, Aeronautical and Maritime Research Laboratories, DSTO, Australia

**10:50 Characterization of Thermal Deterioration of Stainless Steel with Ultrasonic Velocities and Backscattering Noise**—K. Kawashima & S. Ohta, Nagoya Institute of Technology; & T. Isomura, Yahata Steel Works, Japan

**11:10 Nondestructive Inspection of Turbine Blades with Lockin Thermography**—D. Wu, W. Karpen, & G. Busse, University Stuttgart; & G. Zenzinger, Motoren-und Turbinen Union München, Germany

**11:30 Parameter Estimation in Photothermal Measurements with Photodeflection Detection**—R. Bukowski, J. Bodzenta, J. Mazur, & Z. Kleszczewski, Silesian Technical University, Poland

**11:50 Thermoacoustic Vibrometry for In-situ Monitoring of Processes**—J. Stanullo & G. Busse, Universität Stuttgart, Germany; & L. Lyamshev, Russian Academy of Sciences, Russia

**12:10 LUNCH BREAK**

**1:30 POSTERS**  
Room 154

**A Non Destructive Technique, Thermal Wave Imaging to Characterize the Electromigration on Al Alloy**—A. Brun, M. Marty, C. Gounelle, F. Giroux, & H. Roede, Centre Commun CNET-SGS Thomson, France

**Residual Stress Distributions in the Rim of a Steam Turbine Disk Using the  $L_{CR}$  Ultrasonic Technique**—D. Bray, N. Pathak, & M. Srinivasan, Texas A&M University, USA

**Nondestructive Evaluation of Nonconductive Cylindrical Nozzle in Pulsewise Excited Quasi-stationary Electric Field**—Y. Bulbik, Siberian Aerospace Academy, Russia

**Nondestructive Evaluation of Material Parameters Using Neural Networks**—U. Fiedler, M. Kröning, & W. Theiner, Fraunhofer Institute for Nondestructive Testing, Germany

**Nondestructive Thickness Determination of Metallic Coatings Using Ultrasonic Leaky Rayleigh Waves**—J. Coste, F. Lakestani, & W. Vortrefflich, European Commission, Institute for Advanced Materials, Italy

**Nonlinear Ultrasonics for Materials Characterization**—M. Hamilton, Y. Li'inskii, & E. Zabolotskaya, University of Texas at Austin, USA

**Review of Inspection Qualification Programme and RRT Results**—L. Horáček & J. Žd'árek, Nuclear Research Institute Rez, Czech Republic

**Characterization of Metal Surface by Means of Two-Dimensional Fractal Analysis**—S. Horiata, M. Satoh, & H. Kitagawa, Toyohashi University of Technology; & T. Tamiya, Kawatetsu Techno-Research Corporation, Japan

**Tip Location of Exposed and Filled Cracks Using Microwaves**—C. Huber, R. Zoughi, S. Ganchev, & R. Salem, Colorado State University, USA

**Comparison of Parallel Computations with Experimental Visualization of Ultrasonic Waves**—R. Huber, K. Simmonds, R. Schechter, & R. Mignogna, Naval Research Laboratory, USA; & P. Delsanto, Politecnico di Torino, Italy

**Automated Shearography for Measurement of Residual Stresses**—Y. Hung, Oakland University, USA

**Ultrasonic Characterization of Repair Pastes in Context of their Bonds with Metals**—M. Josko, Poznan University of Technology, Poland

**Fractals in Nondestructive Evaluation**—L. Lyamshev, Russian Academy of Sciences, Russia

**Structural Characteristics of Powder Developers: Methods of Measurements**—N. Migoun, Belarussian Academy of Sciences, Belarussia

**On the Matter of Physical Nature of the so called Longitudinal Subsurface Waves**—E. Nesvijski, Protecs Ltd., USA

**Main Physical Characteristics of Liquid Phase Developers**—P. Prokhorenko, A. Kornev, & I. Stoicheva, Belarussian Academy of Sciences, Belarussia

**The Estimation of Elastic Modulus of Metallic Materials By Dynamic Indentation Method**—V. Rudnitsky & V. Djakovich, Belarussian Academy of Sciences, Belarussia

**Ultrasonic Characterization of Burrs in Al-Pressure Castings**—E. Schneider & D. Bruche, Fraunhofer Institute for Nondestructive Testing, Germany

**Ultrasonic Characterization of Texture in Aluminum Rolled Products**—E. Schneider, Fraunhofer Institute for Nondestructive testing, Germany

**Application of Magnetic Barkhausen Effect for Evaluation of Stresses and Structure of Ferromagnets**—V. Vengrinovich, V. Busko, A. Vyshnevsky, & Y. Denkevich, Institute of Applied Physics, Belarussia

## **VII. ACOUSTIC EMISSION AND INTERNAL FRICTION**

**Tuesday, June 20                      Session (A) PM**

*Chairs: P. Maliszewicz, Warsaw Technical University, Poland & M. Rosen, The Johns Hopkins University, USA*

**1:30    Comparison of Absolute Sensitivity Limits of Various Ultrasonic and Vibration Transducers**—C. Fortunko & E. Boltz, NIST, USA

**1:50    Behavior of Concrete Observed by Acoustic Emission Measurement**—P. Maliszewicz, Wroclaw Technical University, Poland

**2:10 Crack Closure During Cyclic Fatigue in Mg-PSZ Ceramic as Detected by Acoustic Emission**—M. Hoffman, TH Darmstadt, Germany; S. Wakayama, Tokyo Metropolitan University; T. Kishi, University of Tokyo, Japan; & Y. Mai & M. Kawahara, University of Sydney, Australia

**2:30 Analysis of the Ultrasound Signal According to the Creep-Resisting Materials Used in Energetics**—F. Cermák, P. Koutník, & F. Kopriva, D-Inspect Service Co., Czech Republic

**2:50 Plastic and Anplastic Behavior of Zirconium Polycrystals**—Z. Trojanová & P. Lukáč, Charles University; & P. Pal-Val, Institute of Low Temperature Physics, Czech Republic

**3:10 BREAK**

**3:30 Stress Relaxation of Short Fibre Reinforced Mg Metal Matrix Composites After Plastic Deformation Due to Thermal Cycling**—J. Kiehn, W. Riehemann, & K. Kainer, Institut für Werkstoffkunde und Werkstofftechnik der TU Clausthal, Germany

**3:50 Detection of Microstructural Changes and Internal Stresses of MMC's by Stress Relaxation Measurements**—W. Riehemann, Institut für Werkstoffkunde und Werkstofftechnik der TU-Clausthal, Germany

**4:10 Low Temperature Internal Friction in Niobium of Different Purity Due to Motion of Geometrical Kinks in Dislocations**—P. Pal-Val, V. Natsik, & L. Pal-Val, B. Verkin, Institute for Low-Temperature Physics and Engineering, Ukraine

**4:30 Acoustoplastic Effects in Crystals**—A. Lebedev, Russian Academy of Sciences, Russia

**4:50 ADJOURN**

## **VIII. ACOUSTIC EMISSION & ACOUSTO-ULTRASONICS**

**Wednesday, June 21 Session (A) AM**

*Chairs: I. Malecki, Institute of Fundamental Technical Research, Poland & O. Taraba, Czech Technical University, Czech Republic*

**8:30 Evaluation of Dilatancy in Rock for a Forecasting of Burst-Prone Zones in Mines**—V. Mansurov, Academy of Sciences of Kirgizstan, Kirgizstan; & V. Anikolenko, Russian Academy of Sciences, Russia

**8:50 Energy - Frequency Distribution of Acoustic Emission from Loaded Rock Samples**—T. Lokajicek & V. Rudajev, Academy of Sciences of the Czech Republic; & R. Prikryl, Institute of Geochemistry, Czech Republic

**9:10 Kinetic Approach to the Nondestructive Monitoring of Rock Failure**—V. Anikolenko, Russian Academy of Sciences, Russia; & V. Mansurov, Kirgizstan Academy of Sciences, Kirgizstan

**9:30 Acoustic Emission in Amorphous Metals**—A. Vinogradov & A. Leksovskiy, Kyoto University, Japan; & A. Ioffe, Russian Academy of Science, Russia

**9:50 Acoustic Emission (AE) As a Tool for Monitoring the Electrical, Thermal and Electromagnetic Effects During the Brittle Cracking of Ceramic Materials**—I. Malecki & J. Ranachowski, Institute of Fundamental Technical Research, Poland

**10:10 BREAK**

**10:30 Acoustic Emission Analysis of Grain Boundary Effect on Plastic Deformation in Bicrystals**—A. Vinogradov, S. Hashimoto, S. Miura, Kyoto University, Japan; & A. Vikarchuk, M. Nadtochiy, Togliatti Polytechnic Institute, Russia

**10:50 Damage Monitoring During Monotonic Tensile Loading of Quasi-Isotropic Carbon/Epoxy Laminates with the Use of Acoustic Emission Technique**—D. Tsamtsakis & M. Wevers, K. U Leuven de Croylaan 2, Belgium

**11:10 Application of Internal Friction and Acoustic Emission Methods for of Machine Manufacturing Materials Properties**—V. Letunovsky, Krasnoyarsk State Technical University; & N. Vasilenko & O. Grigorjeva, Siberian Aerospace Academy, Russia

**11:30 Recent Developments in Real-Time Acousto-Ultrasonic (AU) NDE Technique to Detect & Monitor Various Damage Modes**—A. Tiwari & E. Henneke II, Virginia Polytechnic Inst. & State University, USA

**11:50 Joint-Time-Frequency-Analysis of Acousto-Ultrasonic Waveform Data**—A. Bartos & M. Liang, Computer Sciences Corporation; R. Gewalt, Telos; & T. Gill, Olin Corporation, USA

**12:10 ADJOURN**

## **IX. COMPOSITE MATERIALS**

**Wednesday, June 21 Session (B) AM**

*Chairs: J. Bussiere, National Research Council, Canada & PK Bhagat, Federal Aviation Administration, USA*

**8:30 Computer Simulation of Acoustic Waves Propagation in Elastically Anisotropic Materials**—H. Yamawaki & T. Saito, National Research Institute for Metals, Japan

**8:50 A Study of Lamb Wave Interaction With Defects in Thin Polymer and Metallic Material Using a Differential Fibre-Optic Beam Deflection Technique**—R. Dewhurst & B. Williams, UMIST, UK

**9:10 Observation of Internal Defect in Functionally Gradient PSZ-Ni by Ultrasonic Imaging**—T. Abe & S. Sumi, Tohoku National Industrial Research Institute, Japan

**9:30 Ultrasonic Non-Destructive Testing of the Different Components of the Aircraft Made from the Carbon Fiber Reinforced Plastics**—R. Regazzo, M. Regazzová, J. Válková, & P. Pros, R&R NDT, Czech Republic

**9:50 Study of Interfacial Microstructure in SiC/SiC Continuous Fiber Ceramic Composites by Acoustic Microscopy**—M. Manghnani & V. Askarpour, University of Hawaii, USA

**10:10 BREAK**

**10:30 In Process NDE of Composites for Civil Engineering Applications**—B. Djordjevic, The Johns Hopkins University, USA

**10:50 Potential and Limitations of Microwave NDE Methods for Inspecting Graphite Composites**—R. Zoughi, Colorado State University; & C. Lebowitz, Naval Surface Warfare Center, USA

**11:10 Damping in Magnesium Matrix Composites**—P. Lukáč, Z. Trojanová, Charles University, Czech Republic; & W. Riehemann & B. Mordike, Institut für Werkstoffkunde und Werkstofftechnik, Germany

**11:30 Internal Friction Characterization of Metal Matrix Composites**—L. Parrini & R. Schaller, Institut de Genie Atomique, Switzerland

**11:50 Electrical Resistometry of Mg-Based Microcrystalline Alloys and Mg-Based Composites**—P. Vostry, I. Stulíková, & M. Samatová, Charles University, Czech Republic; & J. Kiehn, K. Kainer, & F. Knoop, Technical University Clausthal, Germany

**12:10 ADJOURN**

## **X. PROCESS CONTROL**

**Thursday, June 22 Session (A) AM**

*Chairs: J. LaPointe, Federal Aviation Administration, USA & M. Hampejs, Skoda Bindery Dept., Bilson, Czech Republic*

**8:30 Analysis of Major and Minor Elements in Gold Jewelry by XRF Modified Proportional Factor Method**—C. Yuanpan & Y. Chongping, China National Nonferrous Metals Industry Corp., China

**8:50 Crystal Growth Rate of Potassium Sulphate for Cooling Crystallization in Ultrasonic Field**—J. Hofmann & V. Roubik, Institute of Chemical Technology, Czech Republic

**9:10 Determination and Monitoring of Through Hole Diameters Using Acoustic Diffraction**—T. Berndt & R. Green, The Johns Hopkins University, USA

**9:30 X-ray Diffraction Applied to Process Monitoring**—C. Ruud, Pennsylvania State University, USA

**9:50 Microwave Dielectric Characterization of Low Density Glass Fibers With Resin Binder**—N. Qaddoumi, S. Ganchev, & R. Zoughi, Colorado State University, USA

**10:10 BREAK**

**10:30 Nondestructive Quality and Process Control in Injection Moulding Polymer Manufacture with Microwaves**—L. Diener & G. Busse, Institut für Kunststoffprüfung und Kunststoffkunde, Universität Stuttgart, Germany

**10:50 Acousto-Ultrasonic Damage Evaluation in Steel-Belted Radial Tires**—H. Reis, University of Illinois, USA

**11:10 Materials Characterization of Powder Metallurgy Products Using Acousto-Ultrasonics**—G. Workman & J. Walker, University of Alabama, USA

**11:30 Process Integrated Nondestructive Testing of Laser-hardened Components**—R. Kern, W. Theiner, & B. Valeske, Fraunhofer Institute for Nondestructive Testing, Germany

**11:50 Nondestructive Characterization of Cure Enhancement by High Power Ultrasound of Carbon Epoxy Composites**—T. Whitney & R. Green, The Johns Hopkins University, USA

**12:10 LUNCH BREAK**

## **XI. PARTICLE TECHNOLOGY**

**Thursday, June 22 - Session (A) PM**

*Chairs: H. Prask, NIST & G. Cariveau,  
NTIAC, USA*

**1:30 Diffraction Techniques in Engineering Applications—K. Kozaczek, C. Hubbard, T. Watkins, X. Wang, & S. Spooner, Oak Ridge National Laboratory, USA**

**1:50 Materials Characterization with Cold Neutrons—H. Prask, National Institute of Standards and Technology, USA**

**2:10 Neutron Depolarization Analysis at Pulsed Neutron Sources for Testing of Micromagnetic Structure and Residual Stresses of Magnetic Layers—L. Chernenko & D. Korneev, Frank Laboratory of Neutron Physics, Russia; & J. Schreiber & W. Theiner, Fraunhofer-Institute for Nondestructive Testing, Germany**

**2:30 Nondestructive Morphological Characterization of Latent and Etched ion Tracks in PETP by Sans—F. Haeussler, M. Hempel, M. Kröning, & H. Baumbach, Fraunhofer Institut fuer zerstörungsfreie Prüfverfahren; & W. Birkholz, Umweltministerium des Landes Mecklenburg-Vorpommern, Germany**

**2:50 Characterization of Microstructure of Plastically Deformed and Thermically Treated Carbon Steel by Means of Positron Annihilation Life Time Spectroscopy in Comparison with Micromagnetic Methods—N. Meyendorf, B. Somiesky & M. Gebner, Fraunhofer Institut fuer zerstörungsfreie Prüfverfahren, Germany**

**3:10 BREAK**

**3:30 Positron Annihilation Measurement in Zr at High Temperatures—R. Král, V. Gröger, & G. Krexner, University of Vienna, Austria**

**3:50 Dislocation Density Measurement and Positron Annihilation—V. Gröger, T. Geringer, W. Pichl, & G. Krexner, Universität Wien, Austria; & F. Becvar, Charles University, Czech Republic**

**4:10 Electron Diffraction Study of Langmuir-Blodgett Lipid Films—L. Orekhova, Institute of Bioorganic Chem.; S. Orekhov, Inst. of Chystallography; & A. Grigoriev, Technical University, Russia**

**4:30 ADJOURN**

## **XII. ULTRASONIC APPLICATIONS**

**Thursday, JUNE 22 Session (B) AM**

*Chairs: R. Livingston, Federal Highway Administration, USA & J. Obraz, President Czech NDT Society, Czech Republic*

**8:30 Nondestructive Materials Characterization for Architectural Conservation—R. Livingston, Federal Highway Administration, USA**

**8:50 Damping of Concrete Beams; Plain, Reinforced and Prestressed—R. Kohoutek, University of Wollongong, Australia**

**9:10 How Calcareous Layers Affect Ultrasonic Thickness Gaging—L. Goglio, & M. Gola, Politecnico di Torino, Italy**

**9:30 Investigation of Spurious Echoes Received in an Ultrasonic Inspection of An Oil Field Tool—D. Bray, W. Tang, B. Bidigare, & L. Cornwell, Texas A&M University, USA**

9:50 **The Evaluation of Integrity of Ceramic-Metal Joints and Ceramic Coatings by C-Mode Acoustic Microscopy**—P. Kauppinen, H. Jeskanen, L. Heikinheimo, M. Siren, & P. Auerkari, Technical Research Centre of Finland, Finland

10:10 **BREAK**

10:30 **Ultrasonic Characterization of Defects in Lead-Magnesium Niobate (PMN) Smart Materials**—J. Bernstein, J. Wagner, & J. Spicer, The Johns Hopkins University, USA

10:50 **Measurement of Adhesion Strength Using Nonlinear Acoustics**—S. Pangraz, M. Kröning, & W. Arnold, Faunhofer Institute for NDT, Germany

11:10 **On-line Ultrasonic Testing System of the Next Generation by Using Real-Time Chirp Pulse Compression Processing**—M. Yoshino, R. Okuno, A. Nagamune, & K. Nishifuji, NKK Corp., Japan

11:30 **An Automatic Ultrasonic Testing System for the Butt Weld Zone of the Gas Pipe Line**—H. Yamada, H. Yamaji, T. Hyoguchi, & T. Udagawa, Nippon Steel Corp., Japan

11:50 **The Ultrasonic Testing of Welding in Plastics**—N. Gil, G. Konovalov, A. Mayorov, P. Prokhorenko, Belarussian Academy of Sciences, Belarussia

12:10 **LUNCH**

### **XIII. BASIC ULTRASONICS**

**Thursday, June 22                      Session (B) PM**

*Chairs: C. Fortunko, NIST, USA & L. Lyamshev, Russian Academy of Sciences, Russia*

1:30 **Determination of the Elastic Constants of Anisotropic Solids With an Artificial Neural Network**—R. Sribar, General Electric Co.; & W. Sachse, Cornell University, USA

1:50 **Negative Elastic Constants in Intermediate Valent  $\text{Sm}_x\text{La}_{1-x}\text{S}$** —U. Schärer & P. Wachter, ETH, Switzerland

2:10 **Estimation of Parametric Models for Double Transmission Experiments on a Viscoelastic Plate**—D. Zhou, L. Peirlinckx, & L. Van Biesen, Vrije Universiteit Brussel, Belgium

2:30 **Strategy Towards Nondestructive Evaluation of Mechanical Properties of Steel Components**—E. Schneider, W. Theiner, & M. Kroning, Fraunhofer Institute for NDT, Germany

2:50 **Internal Friction in Magnesium Alloys Prepared by Rapid Solidification**—Z. Trojanová & P. Lukáč, Charles University, Czech Republic; & S. Kraft & W. Riehemann, & B. Mordike, Institut für Werkstoffkunde und Werkstofftechnik, Germany

3:10 **BREAK**

3:30 **Qualifying Indentation Fracture Toughness Testing by Ultrasonics**—F. Bergner, Institut für Werkstoffwissenschaft; & B. Köhler, Fraunhofer Einrichtung für akustische Diagnostik und Qualitätssicherung, Germany

3:50 **Nonlinear Acoustic Parameter and Strength of Solids**—W. Wu & F. Han, Nanjing University, China



**4:10 New Digital Techniques for  
Precise Measurement of Surface Wave  
Velocity With an Acoustic Microscope—M.  
Okade, T. Hasebe, & T. Kawai, Aisin Seiki  
Co., Ltd., & K. Kawashima, Nagoya Institute  
of Technology, Japan**

**4:30 Estimation of Ultrasonic Source  
Distributions of Electroacoustic  
Transducers—D. Zhou, L. Peirlinckx, M.  
Lumori, & L. Van Biesen, Vrije Universiteit  
Brussel, Belgium**

**4:50 A New Approach to Ultrasonic  
Image Reconstruction—M. Yamano,  
Sumitomo Metal Industries, Japan; & S.  
Ghorayeb, Iowa State University, USA**

**5:10 Waveform Mapping of  
Piezoelectric Transducer Impulse  
Responses in Multi-Transducer Pattern  
Recognition-Based UNDE Systems—A.  
Bartos, M. Liang, & T. Lyon, Computer  
Sciences Corporation; & T. Gill, Olin  
Corporation, USA**

**5:30 ADJOURN**

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## **EARLY RECOGNITION OF H-INDUCED STRESS CORROSION CRACKING WITH MICROMAGNETIC TESTING METHODS**

M. Lang & I. Altpeter, Fraunhofer-Institute for Nondestructive Testing, Germany

Hydrogen-induced stress corrosion cracking in ferritic steels of high strength can lead to sudden failure of a component and is thus a safety risk. This is especially important for the production, transportation and processing of mineral oil, e.g. for pipelines, oil tankers, oil refineries or desulphurization plants. The hydrogen absorbed by steel leads to tensile residual stresses which interact with the mechanical load and favour stress corrosion cracking.

The present study relates to the fundamental development of a nondestructive testing method which allows the detection and interpretation of hydrogen absorption at an early stage, i.e. before crack formation commences. The development of the nondestructive method is based on the interaction of magnetic Bloch walls and Bloch wall structures with second or third order microstructural and residual stress changes causing stress corrosion cracking. Here was made use of micromagnetic testing parameters such as the magnetic Barkhausen noise, coercivity and the distortion factor.

The investigations were carried out on martensitic and tempered martensitic structures of the steel grade X 20 Cr 13 (material no. 1.4021) in H<sub>2</sub>S-saturated NaCl solution, pH 3 (NACE solution), as corrosion medium.

It has been possible to show that the process developed is substantially more sensitive to the hydrogen-induced deterioration of materials than are conventional nondestructive methods such as ultrasonic, eddy current and magnetic particle inspection.

## **THE DEVELOPMENT OF NONDESTRUCTIVE EVALUATION (NDE) FOR MONITORING THE EMBRITTLEMENT IN NUCLEAR REACTOR PRESSURE VESSELS**

M. Blaszkiewicz, Westinghouse Science and Technology Center, USA

Irradiation induced degradation of light water reactor pressure vessels, known as embrittlement, is of primary concern to operating nuclear power plants facing the possibility of being shut down before their license expiration date. Currently, the degree of embrittlement is determined using approved models and guidelines. Reactor vessel surveillance programs provide further information about the condition of the vessel through mechanical testing of pressure vessel material samples removed from surveillance capsules. However, the models and surveillance programs do not always provide enough accurate information to support decisions to end life prematurely, to continue life until the license expiration, or to extend life past the original design using the annealing process. In addition, the effects of annealing and re-embrittlement on the vessel integrity have not been

adequately addressed by the models and surveillance programs. Mechanical tests, such as Charpy and tensile tests, used to establish the level of embrittlement, are dependent on the intrinsic properties of the material, such as precipitate size and concentration, and dislocation density. NDE options dependent on intrinsic material properties are being explored so that embrittlement in reactor pressure vessels can be assessed using nonintrusive methods. Electrical, magnetic, electromagnetic, ultrasonic, and micromechanical techniques have been investigated for use in detecting changes in microstructure of pressure vessel steels or related materials. The various techniques and corresponding results are reviewed, and results of present investigations are given. It is shown that there is a need for correlation not only between the microstructural changes and the NDE results, but also between the NDE results and the mechanical behavior or level of embrittlement. It is hoped that the more common, but also more costly, destructive techniques of tensile and Charpy testing of surveillance capsule specimens will be augmented, or even replaced, by cost-effective, *in situ*, and possibly on-line NDE techniques.

## **EVALUATION OF DIGITIZED SIGNALS FROM DEFECTOSCOPIC CHECKING OF STEEL ROPES**

O. Lesnák, Research Mining Institute, Czech Republic

The automated processing and evaluation of measured technical values by means of the up-to-date computer technics has been recognized as a very fast developing technical discipline. These method of working should be a basis of automated control and automated production, as well. There is also fully acceptable that the development has to be in touch with the defectoscopy tasks solutions, mainly in the field of steel ropes defectoscopy.

Recently, some necessary preconditions have been created as to this activities, i.e. the first defectoscopic equipment using the digitalized output of measured values type MID-5H and MID-5HVS were designed. There should be considered as an usual logical step that some up-to-dated computer aided equipment for visualization, evaluation and processing of measured digitalized values has to be designed aimed at the fully automated processing of the ones.

The paper discuss the baseline philosophy of visualization of digitized data which has been firstly used in cases of defectoscopic checking of steel ropes. A method of visualization of the very data using the wide as well as narrow coil and/or equipment with Hall's probes is also described.

Prime advantages of the signal digitization are as follows:

- possibility of automation of measured data processing and evaluation,
- enhancing of the information quality of the used method.

Technical advantages are as follows:

- More precise recording of measured data,

- recording of measured data on the magnetic medii with possibility of further processing with the equal quality as in case of first processing,
- in course of the first visualization, i.e. during the measurements (system on-line), there is possible to visualize pre-processed data,
- the measured data record could be analyzed using various horizontal and vertical scales,
- there is possible a graphical comparison of two or more data packages measured on the individual rope, i.e. in the defects channel and corrosion one, as well,
- processing and evaluation of measured data by means of mathematical and statistical methods,
- possibility of transmission of measured data without any losses on long and very long distances.

The paper deals also with problems on necessary computer technics specification and some further tends on processing of measured digital data aimed at better information ability of the method used.

## **DETECTION OF VARIATIONS IN HEAT TREATMENT AND CONDUCTIVITY IN METALS USING SURFACE MAGNETIC FIELD MEASUREMENT TECHNIQUE**

D. Mirshekar-Syahkal & R.F. Mostafavi, University of Essex, United Kingdom

Surface magnetic field measurement technique (SMFM) is a simple powerful electromagnetic technique for detecting and sizing surface breaking cracks in metals. In some respects, this technique resembles the thin skin eddy current method. However, unlike the latter, it does not rely on the measurement of impedance. It also differs from the ac flux leakage method in that it does not exploit the magnetic flux, leaking from the crack opening. Furthermore, unlike a similar method known as the ac field measurement technique, the SMFM technique does not use eddy currents with uniform current distributions at the metal surface. The SMFM technique is based on the measurement of the magnetic field component parallel to the metal surface, using a magnetic sensor such as a small coil or a tape-head probe. The magnetic field is produced by the induction of a high frequency eddy current in the metal. For this purpose, a set of appropriately shaped current-carrying wires is located above the workpiece. In practice, the probe and the current inducer are attached together in a particular arrangement. There are many useful probe-inducer combinations, each having its own properties as far as flaw detection and characterization are concerned.

Although the SMFM technique was originally developed to detect surface breaking cracks in metals, our recent experiments showed that it is capable of reliably detecting variations in heat treatment and conductivity in metals. Detection of these variations are of significant importance in the industry for estimating the susceptibility of a metal to

cracking. Usually such variations are small and for example, the eddy current detection requires careful measurements using sensitive equipment. To achieve high sensitivity in the SMFM technique without increasing the complexity of the detecting systems, a special combination of the probe and the inducer has been developed.

In this paper, the principles behind the SMFM technique are briefly reviewed and the new probe-inducer arrangement for applications requiring high sensitivity is introduced and discussed. The paper, then, presents the results of the measurements taken on aluminum containing heat affected zones of different severity. It also examines the results of variations in the conductivity in aluminum due to impurities. The sample used for this experiment is a block of aluminum with cylindrical brass inclusions in its surface.

## **FERROMAGNETIC SURFACE LAYERS TESTING WITH DEPTH RESOLUTION USING A PRIORI KNOWLEDGE**

V. Vengrinovich & S. Zolotarev, Belarussian Academy of Sciences, Belarussia

It is well known, that surface layers after heat treatment, cold rolling or other type of strengthening exhibit strong structure or stress inhomogeneity. At the same time after their nondestructive testing we are interesting not in properties, averaged over the whole layer, but in their depth distribution. For this reason it is necessary to apply the real time mathematical reconstruction technique, and this problem is usually the problem of ill-posed inversion. In order to receive stable solutions of these equations which minimal square root errors it is necessary to use all a priori information which is known about the object under testing.

The account of this a priori knowledge could be provided at the stage of physical simulation of the object [1] as well as during the solution procedure by means of extraction the solution, which satisfies to some previously known properties (equilibrium condition, zero equalization of the result on some edges of the body, zero equalization of the derivatives etc.). One of possible ways for accounting this information was considered in [2]. In this report we integrate this approaches. In general the surface layers' testing with depth resolution gives qualitatively new information which can't be received by other techniques.

1. V. Vengrinovich, S. Zolotarev. *Rus. J. Nondestructive testing*, No. 4, 1994, pp. 40-43.
2. V. Vengrinovich, S. Zolotarev. *J. of Technical diagnostics and NDT*, No. 3, 1992, pp. 14-18.

## **BARKHAUSEN ANALYSIS OF THE EFFECT OF STRAIN AND HEAT TREATMENT ON EPSILON-MARTENSITE**

I. Mészáros & M. Káldor, Technical University of Budapest, Hungary

Barkhausen noise (BN) is generated by the discontinuous motion of Bloch walls induced by changing external magnetic field. The BN allows to characterize the amount and the microstructural state of magnetic components of materials. The aim of the work is to study the influence of cold work and heat treatment on the microstructure, on the magnetic BN and their correlation with mechanical properties. Magnetic measurements have been made to characterize the amount of strain induced epsilon-martensite in cold worked (18%Cr, 8%Ni) alloyed austenitic stainless steel. The epsilon-martensite produced by plastic deformation appears inside the austenite grains within slipping plans in the form of stacking faults and twins. It has hexagonal crystal structure and it is the only ferromagnetic component of the low carbon austenitic stainless steels.

The stainless steel specimens were cold worked at room temperature up to about 50% strain. The microstructure was examined by BN energy-, saturation induction measurement and by optical microscopy. The ratio of the para and ferromagnetic phases was controlled by Mössbauer-spectroscopy. The results were compared to hardness measurement data. The energy of BN were calculated from the power spectra of the noise - obtained by Fourier transformation of the time signal- was integrated in the 0.3-38 kHz frequency range.

It was found that the BN energy increases rapidly with the increasing deformation in the 0-50% strain range according to the increasing amount of epsilon-martensite. The method was found to be very sensitive and quantitative measurement to identify the amount of strain induced martensite.

In the second part of the present work the uniformly elongated (40%) specimens were heat treated isocronically (for 30 minutes) in the 100-1000 °C temperature range. The BN energy and the saturation induction started to decrease at about 320 °C and at 600 °C reached a very low level which corresponds to the non deformed state while the hardness practically remained at its original level. The annealing process which caused the significant decrease of hardness started at 650 °C. Although the increase of both the hardness and BN energy must be caused by the increasing amount of epsilon-martensite during the plastic deformation the magnetic and hardness recovery processes started at significantly different temperatures. The amount of the epsilon-martensite phase did not change below 650 °C temperature according to the saturation induction, Mössbauer spectroscopic and hardness measurement results.

We suppose that the microstructure of the epsilon-martensite has changed during the heat treatment process. The interesting microstructural change of martensite is explained by the clustering of carbon atoms and precipitation of complex (Cr, Ni) carbides

respectively within the martensitic region. The relatively small martensitic volumes are supplied with carbon from the surrounding high volume austenitic parts of the grains by very high rate surface diffusion process. Although the carbon content of the austenite grains is low (less than 0.1 wt.%) the carbon concentration of martensitic regions can reach high levels because of the large volume difference between them. The carbide precipitates are impassable obstacles for the domain walls. Consequently the carbides delay and prevent the movement of domain walls respectively which cause the decrease of BN energy.

The suggested way of BN measurement is very useful in detection of magnetic phase in a paramagnetic phase and an easy nondestructive way to characterize the deformation related damage (fatigue damage) at room temperature. The method is usable for not only stainless steels but for all steels which contain metastable austenite from which the damage process or the plastic deformation can produce ferromagnetic epsilon--martensite.

### **PECULIARITIES OF CONNECTION BETWEEN MECHANICAL PROPERTIES AND RESIDUAL MAGNETIZATION OF ARTICLES OF DIFFERENT SIZE**

S.G. Sandomirskii, Belarussian Academy of Sciences, Belarussia

The heat treatment (hardening, tempering, annealing) of steel and iron articles is realized to receive the mechanical properties (hardness, strength limit, relative elongation and so on) are required. The inadmissible changes of mechanical properties for articles appear due to the different possible changes of temperature and time regimes of heat treatment. The only nondestructive testing it is possible if anyone needs to test the mechanical properties of all the produced articles. The magnetic method is most preferable method of nondestructive testing for mechanical properties of ferromagnetic articles. The correlation between the mechanical properties under testing for articles and their magnetic characteristics (coercive force  $H_c$ , residual magnetization  $J_r$ , saturation magnetization  $J_s$ ) is the physical basis of the magnetic method of testing. The magnetic method allows to automatize the process of testing completely. The automatized devices for testing the moving articles are developed. The articles are magnetized when they are in free fall through the magnetic field created by the coil with a current or the permanent magnets. When the articles continue moving and fall through the region without magnetic field the residual magnetization of article  $J_d$  is measuring. The parameter of article (demagnetization factor  $N$ ). The analytical expressions for sensitivity of  $J_d$  to  $H_c$ ,  $J_r$ ,  $J_s$  and  $N$  were established as a result of theoretical and experimental investigations. This allows to predict the influence of mechanical properties of articles on  $J_d$  by using the known correlations between mechanical and magnetic properties. The most interesting results are when  $H_c$  increases and  $J_r$  decreases during the changing of hardness for material. In this case  $J_r$  for short articles (large  $N$ ) increases and for long ones (small  $N$ ) decreases when hardness increases.



## **NON-DESTRUCTIVE MEASUREMENT OF GRAIN SIZE IN STEEL PLATE BY USING MAGNETIC COERCIVE FORCE**

M. Yoshino & H. Tanabe, NKK Corp.; T. Sakamoto, Sumitomo Metal Industries Ltd.;  
N. Suzuki, Kobe Steel Ltd.; & Y. Yaji, Nippon Steel Corp., Japan

The characterization of micro-structure of steels by nondestructive techniques has become increasingly important in recent years. In-line/on-site measurement of structural properties such as grain size, residual stress and formability is essential for optimizing manufacturing process and predicting the final properties.

Various measurement techniques using magnetic properties have been developed to characterize micro-structure of steels, but magnetic properties depend on not only a interested variable but also on many other variables. Therefore, in grain size measurement by using magnetic properties, the effects of other variables like residual stress, metal structure must be evaluated quantitatively. A round robin test, which was promoted by the Committee on Sensors for Micro-structure of the Iron and Steel Institute of Japan (ISIJ), had been carried out for about three years.

First, in our round robin test, a set of ring specimens of low-carbon steel with different grain size, in which ferrite phase dominated over other phases was used. The results showed that the coercive force had the strongest correlation with grain size among the magnetic properties. Consequently, the coercive force was chosen for measuring grain size.

Second, grain sizes of various plate specimens with variation in terms of residual stress, surface finish, volume fraction of secondary phases, etc. were measured by using the coercive force. The experiment gave the following results.

- 1) A lower freq. about 0.005Hz for the magnetic excitation brought much better correlation than 0.1Hz, because of the skin effect.
- 2) The grain size of the limited specimens of the low-carbon steel with under 17% pearlite and no martensite phase, even if they have different residual stress and grain size distribution, was estimated with fluctuations of  $\pm 1$  in ASTM class.
- 3) The amount of secondary phases (such as perlite, martensite) increased the coercive force, especially when pearlite was over 60% and martensite over 15%.

## **DETECTION OF THE TENDENCY TO CHILLING IN SERIES MANUFACTURED CAST IRON COMPONENTS USING MICROMAGNETIC TESTING PROCEDURES**

M. Kröning & I. Altpeter, Fraunhofer-Institute for Nondestructive Testing; &  
U. Laub Q NET GmbH, Germany

For the last decades cast iron producers have been engaged in the problem of tendency to chilling in cast iron components. Tendency to chilling means that there is an unintended appearance of ledeburite and cementite phases in cast iron that normally congeals as grey cast iron. This causes a decrease of ductility resulting in safety problems and furthermore a local increase of hardness leading to the destruction of machining tools.

Micromagnetic testing procedures like *magnetic Barkhausen noise* and the *analysis of the higher harmonics of the tangential field strength* are used for the nondestructive detection of the tendency to chilling. These micromagnetic testing procedures use the interaction between microstructure states and remagnetization behavior for material characterization. Measurements on various components have demonstrated the detectability of chilled microstructure states showing a good correspondence with metallographic results received at the same location as the micromagnetic results. Thus the nondestructive test can replace the random selection of test specimen for destructive tests (SPC) and can be applied on-line in a closed loop control.

## **NONDESTRUCTIVE DETERMINATION OF ELASTIC INTO THE MICROPLASTIC STATE TRANSITION**

L. Keller & P. Stanek, TSI System s.r.o., Military Technical Institute of Protection,  
Czech Republic

The transition from the elastic into the plastic state of metallic materials in the case of a typical stress-strain diagram at the static tensile test is given by specific stress being characteristic for mechanical properties of the material. The elastic limit is the stress at which the permanent elongation remains 0.005% after unloading of the specimen. The proportional limit is the stress at which the change of the tangent direction to the curve of the diagram may still be neglected. The yield stress represents the stress at which the specimen will be considerably prolonged without increase in stress. If this cannot be seen in the diagram instead of this the proof stress is determined which is the stress which causes permanent elongation by 0.2%.

Determination of a physically justified stress at which the material passes from the elastic into plastic, or microplastic state is of a special importance at the selection and calculation of materials for parts of highly stressed products, e.g.: Springs, bearings, wire cords, barrels etc. Where a high resistance against microplastic and small plastic deformations is required.

It is known that in the case of many monocrystals the elasticity limit is clearly expressed under which neither residual deformation nor flexible hysteresis exist. Based on experimental research of polycrystals it can be estimated that there is an elasticity limit close to the absolute one which can be named as a elasticity threshold. In the case of stress under this elasticity threshold residual deformation does not occur even if the measurement accuracy is increased.

In this context an important role have test methods based on the response of the state of the material structure in its electromagnetic characteristics and at loading of test pieces by tensile test. The coupling of the testing instrument with the tested material in the shape of a long cylindrical tensile test specimen is provided by the encircling coil based on electromagnetic induction. The measuring signal of the magnetic hysteresis loop is transformed to the Fourier coefficients being used as descriptors of the deformation characteristics. They determine the yield point in the course of the tensile test and especially the unelastic limit which is deep under the yield point. This limit cannot be determined from the stress-strain diagram in the case of such small deformations. Unelastic limit is given by percolation transfer caused by the interaction of Weiss domains for a long distance under the influence of increasing dislocation density due to the increase in the specimen load. The higher this limit will be the more resistant the tested material will be against damage. That is why this test seems to be reasonable for testing the quality of the material used for parts of highly stressed products. The paper will deal with the testing method description as well as with results of experiments performed.

## **OVERVIEW OF MICROWAVE NDE APPLIED TO THICK COMPOSITES**

R. Zoughi & S. Ganchev, Colorado State University & G. Carriveau, NTIAC-TRI/; USA

Application of composite materials in a wide variety of areas continues to grow at a high rate. Advanced engineering and manufacturing approaches have promoted composite uses when thick section components are required. These strong, light-weight materials offer many benefits over traditional monolithic materials. However, they also present a significant challenge when nondestructive evaluation methods are applied. NDE difficulties arise from inherent composite material properties, for example, anisotropy, inhomogeneity, and acceptable flaws and defects resulting from manufacturing or induced in service. In addition, most thick composite materials are highly absorbing and/or scattering to traditional NDE energy probes such as heat, sound, x-rays, etc. Microwave NDE techniques offer some novel solutions for the inspection and evaluation of thick dielectric composites. This paper will present an overview of microwave NDE applications for these thick materials, describing theoretical and experimental results from materials ranging in thickness from one centimeter to over 10 centimeters. The experiments were performed on well characterized standard materials containing intentionally introduced flaws and defects including: holes/voids, delamination/disbonds, contaminating materials, and impact damage. Comparison of microwave NDE results will be made with other NDE methods such as ultrasonics, radiography, thermal imaging, and optical methods using the same standards.

## **CHARACTERIZATION OF GREEN CERAMICS BY MICROWAVES AND ULTRASOUND**

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Quality assurance in ceramics production is most efficient, if sources of failure are detected at a very early stage of the manufacturing process. The present tendency of machining green ceramics to a near-final shape before sintering requires a good knowledge of inhomogeneities of the green body in order to keep deformation and generation of internal stresses during sintering under control.

In this contribution, volume properties of green ceramic samples of alumina and other ceramics are characterized by microwaves in the 75 to 100 GHz range on one hand and by ultrasound measurements at up to 2 MHz on the other hand. Volume properties are determined by careful time-of-flight measurements. For ultrasound, the transmission time for short pulses is measured directly. Dry coupling transducers designed for green ceramics application are employed. For the microwave experiment, determination of the complex transmission coefficients over the frequency range available and a subsequent Fourier transform give the time-of-flight with a resolution of about 0.3 ps. Useful information is extracted from a comparison of ultrasound and microwave data, as different physical mechanisms are involved. For microwaves, time-of-flight is determined by  $\sqrt{\epsilon}$  where  $\epsilon$  is the real part of the effective dielectric constant, whereas for ultrasound time-of-flight is governed by  $(p/M)^{0.5}$ , where  $p$  is the average density and  $M$  an elastic modulus.

We have performed time-of-flight measurements as a function of the position on the green ceramic specimens. Our results obtained on green state alumina cylinders show that velocity variations of up to 0.7% can be observed at different positions of the samples by microwaves, at the same time a variation of up to 8% is found by ultrasound. The ultrasound and microwave velocities are inversely correlated along the test tracks. A course explanation is that a higher compaction density results in a higher ultrasound velocity and in a higher dielectric constant, thus in a lower microwave velocity. Present investigations try to attribute these results in more detail to the powder and binder concentrations.

## **NON-DESTRUCTIVE MOISTURE MEASUREMENT USING MICROWAVES**

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Although there are a variety of moisture measurement methods, microwave techniques are attractive since they offer nondestructive, on-line measurements. These are desirable in many processing industries, including, for example, those associated with power, chemical commodities, construction and food/agriculture. Over the past few years,

the cost of microwave components has been significantly reduced owing to the increased use of these components in consumer items and therefore it has been possible to fabricate instrumentation at an acceptable cost and of a rugged form suitable for making microwave measurements on industrial plant. The basic theory of permittivity of moist materials will be given and relationships between the real and imaginary permittivity and measured parameters such as attenuation and phase shift will be developed. Cole-cole graphs will be introduced to show how sample permittivity has a strong dependence on temperature. Other effects, such as density, particle size and anisotropy will be discussed together with the nature of the binding of the water. Experiences of operation of several instruments will be presented:

- The Infrared Moisturex microwave paper meter
- The QPar Angus stripline probe
- The Hydronix sand/cement hydroview sensor

With these experiences (listed above) it is hoped to show that the microwave method does, indeed, offer a unique niche amongst the moisture measurement methods.

## **DEFECT CHARACTERIZATION BY A MICROWAVE TESTING SYSTEM AT 30 GHZ COMPARED WITH RESULTS OF OTHER NDE-METHODS**

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Early and reliable detection of defects is of vital interest for quality control, and various established NDE methods are being widely used. This paper deals primarily with NDE results obtained using non-destructive microwave raster scan imaging performed with an open ended waveguide system at 30 GHz. Various polymer and wood samples with 2- and 3-dimensional defects and structures are investigated. We will discuss how lateral resolution and defect characterization depend on depth underneath the sample surface.

The direct comparison with other NDE-methods as ultrasonics, lockin-thermography and x-ray reveals the specific potential of the microwave techniques. On this background one can optimize the application of NDE methods with respect to the kind of sample and the kind of defect to be analyzed.

## **MODELS FOR MICROWAVE NONDESTRUCTIVE TESTING OF MATERIALS**

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The need for testing of dielectric and lossy dielectric materials has renewed the interest in high frequency methods of testing. In particular, testing with microwaves and millimeter waves has received new attention because of their suitability to work with

nonmetallic composite materials. This renewed interest presents new challenges in modeling and characterization of these complex materials. The common models used for high frequency applications such as the method of moments cannot, in general, be used for this purpose, primarily because they cannot take into account sources but, perhaps more importantly, because they have been developed specifically as "far field" models. On the other hand, finite elements, and combined finite elements-method of moments methods can, and are being used for accurate and detailed modeling of the testing environment.

This paper presents some useful techniques, applicable to the test environment at all frequencies, but in particular in the microwave and millimeter wave domain. The techniques are based on the finite element method, derived from the Huygens principle. Anticipating both testing in resonant cavities and non resonant closed structures as well as scattering methods, two separate, broad techniques have been devised for this purpose.

One method, suitable for resonant structures is based on evaluation of resonant frequencies of the system using an electric field formulation. The method can be used for modeling of lossless and lossy dielectrics as well as monitoring of production processes in microwave cavities.

The second method is a scattering method suitable for modeling in open domains and in the vicinity of apertures. Both lossy and lossless dielectric materials can be modeled. The methods are described and representative results are given to demonstrate their utility in modeling microwave nondestructive testing processes.

## **ACOUSTICS OF WOOD**

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The acoustics of wood is related to three main topics:

- environmental acoustics, related to the acoustics of forests and acoustic quality of some forest products or to the utilization of wood and wood based material in architectural acoustics.
- material characterization, make reference to the theory and experimental methods for the elastic characterization of wood. Elastic constants for solid wood and for wood based materials can be deduced by ultrasonic techniques. Structural features of wood are related to ultrasonic parameters.

- quality assessment of wood products, considers the acoustical properties of wood species for musical instruments, the methods for nondestructive control of trees, timber and wood composites, the defects detection, the method of acoustic emission, the high energy ultrasonic treatment for wood processing.

### **NONDESTRUCTIVE EVALUATION OF LOGS FOR STRUCTURAL PRODUCT QUALITY**

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Past nondestructive evaluation efforts have paved the way for the successful use of NDE for determining the quality of finished wood products. Little effort has been expended, however, on developing NDE techniques for use in grading or sorting logs for structural quality. The USDA Forest Products Laboratory has recently conducted a series of studies to address this deficiency. This presentation will present results from these studies.

Longitudinal stress wave NDE techniques were used to evaluate the quality of approximately two hundred balsam fir and white spruce logs prior to processing into lumber. Longitudinal speed of sound transmission was determined for each log. The modulus of elasticity of each piece of structural lumber from the logs was then determined using transverse vibration NDE techniques. A strong relationship was observed between the modulus of elasticity of the logs determined from the stress wave NDE and the modulus of elasticity of the lumber obtained from them.

### **VIBRATIONS OF PIANO SOUNDBOARDS - REAL SOUNDBOARD WITHOUT RIBS IN COMPARISON WITH ITS FEM MODEL**

J. Skala & A. Raffaj, PETROF Piano Factory, Czech Republic

The numerical modelling of the piano soundboard vibrations and board sound radiation is very effective for new instruments construction. In the beginning we have been coming from simple models (a rectangular lath of wood) to complicated (consisting from the resonance plate with the ribs and the bridges), checking back results of finite elements method models by experimental model analysis. Some differences were found. We have tried to determine the portion of model behavior dispersion originated by the inaccuracy of input elastic constants measurement. This contribution is comparing some methods for obtaining elastic constants of spruce resonant wood. That are method of static tension, resonant bending, ultrasonic for longitudinal waves, non resonant impedance measurement, resonant impedance measurement and tensiometric one. At first, the systematic error of measure elastic constants was followed up. This error originate from invalidity of theoretical assumptions. At the second we followed up the magnitude of

measured values dispersion. The reasons of the dispersions are variability of material of specimen and inaccuracy and noise in sensing main variable. At the third we compared degree of acceptability and applicability of measure methods to our purpose.

## **part II**

The upright piano soundboard is made up of spruce wood and angel of grain and soundboard edges is approximately  $45^\circ$ . We can consider upright piano soundboard as very thin generally orthotropical plate. "Generally" means grain are not parallel with edges. Spruce wood possess approximately orthotropic symmetry.

Different upright piano soundboards with free edges were analyzed by experimental modal analysis (EMA). Modal frequencies and mode shapes of different ribless soundboards are obtained. Afterwards these soundboards have been cutting to form of specially orthotropical plate means grain ran parallel with plate edges. The linear vibrational properties of such "sheets" are governed by four elastic constants. All four elastic constants have been determined from measurement of resonant frequencies of low-frequency modes of these rectangular plates with free edges.

These constants were used as input parameters to FEM model of soundboard. Computed frequency and mode shapes are compared to experimental obtained ones. Sensitivities of all computer model input parameters are investigated.

## **DURABILITY ASSESSMENT OF POLYMER MATRIX COMPOSITE MATERIALS**

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This paper describes a novel approach to the characterization of properties and the assessment of long-term durability of polymer matrix composite materials. Our laboratory has developed "intelligent" robotic work cells and nondestructive testing techniques to increase laboratory productivity and improve the quality of test information needed to assess environmental durability and to guide the specification, design and manufacture of composite materials. This approach facilitates measuring chemical, physical and mechanical property changes of many different composite specimens under a wide variety of accelerated environmental exposure conditions (time, temperature, humidity, light, mechanical stress, and recycling). Advanced computing and expert system technologies are employed to facilitate real-time monitoring, control and integration of the robotic work cells; planning and scheduling tests; and automating data/knowledge acquisition and analysis. The robots automatically handle test specimens, operate equipment, and conduct tests. Some tests, such as measuring water absorption and changes in the dimensions of individual specimens, require not only a high degree of robot dexterity, but also flexibility in sequencing and integrating operations. Results of experimental studies on the durability of an epoxy resin/glass fiber reinforced composite material are presented to demonstrate the



advantages of combining "intelligent" robotics with nondestructive techniques for measuring property changes. For example, diffusion constants and associated thermo-dynamic parameters determined from water absorption measurements at different temperatures combined with data obtained from nondestructive dynamic mechanical measurements of laboratory specimens are useful in predicting the effects of long-term environmental exposure. Moisture expansion, specimen thickness, and fiber-orientation effects are evaluated. Video imaging and digital image analysis techniques provide complementary visual and quantitative information about microvoid formation and fracture damage.

### MEASUREMENT OF DISLOCATION DENSITY BY RESIDUAL ELECTRICAL RESISTIVITY

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In the last years, the *residual electrical resistivity* has been closely re-inspected for its use to measure dislocation densities [1], with the motivation to establish a nondestructive, simple and global analysis technique, in contrast to widely used Transmission Electron Microscopy (TEM). It turned out that the method can be applied in a simple way up to dislocation densities  $N$  of order  $\approx 10^{14} \text{ cm}^{-2}$  which exceeds the upper limit of TEM by about 4 orders of magnitude while the measuring accuracy  $\Delta N$  is of order  $\approx 10^9 \text{ cm}^{-2}$  being about the same as provided by TEM. The reliability of the resistivity method is confirmed by quite satisfactory coincidence with dislocation density measurements by other techniques like TEM, calorimetry [2] and x-ray line profile analysis [3].

Although the method can be performed in a comparably easy manner, it requires certain procedures to account carefully for all defects which were either present before the dislocations to be measured, or generated together with dislocations (e.g. point defects in plastic deformation). These procedures are discussed in detail by the present paper.

In principle, the measuring accuracy could be enhanced far beyond the value given above (i.e. by modern Lock-In and SQUID techniques); however, some problems arise with measurement of *low* dislocation densities  $N \leq 5 \times 10^9 \text{ cm}^{-2}$  where the dislocation resistivity no longer exceeds that of impurities even in comparably pure metals so that considerable Deviations from Mattiessen's Rule (DMR) occur. At least for the noble metals, however, these contributions can be correctly quantified [4,5] and thus be taken into account for calculation of the true dislocation resistivity. Larger problems may arise when the dislocations are pinned by certain impurities in positions with high internal stress level: Here not only additional resistivity measurements of low energy dislocation arrangement (checked by TEM) would be required but also those of Low Field Hall Coefficient in order to quantify the related DMR - contribution.

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### **ANISOTROPY OF YOUNG'S MODULUS AND TECHNOLOGICAL PROPERTIES**

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Technological properties dependent on mechanical properties are in standards usually characterized by yield strength, ductility and hardness of given material. Problems due to fluctuations of technological properties of spring material manufactured in the form of bands for electronic industry revealed gaps in used standard.

Unrevealed changes in technological properties of tested brass band used in electronic industry for connector spring tangs resulted in twisting of the strip with connector tangs. This twisting has been intuitively explained by anisotropy in distribution of residual stress after rolling the band and cutting the strip with connector tangs. X-ray measurement of texture in brass band proved fluctuation in anisotropy of elastic moduli due to human factor affecting the technology of rolling the copper alloy sheet used for manufacturing bands and connector tangs. Results support idea the role of the preferred orientation of metallurgical structure for technological properties of metal plate has been underestimated.

### **RESIDUAL STRESS DEPTH PROFILES OF AUSROLLED 9310 GEAR STEEL**

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Oak Ridge National Laboratory, USA

Residual Stress analysis utilizing x-ray diffraction in conjunction with material removal by chemical polishing provides a very effective method of analyzing the near surface residual stress profile of steels. In this experiment, residual stress profiling has been used to analyze the effects of surface ausrolling during the marquenching of a 9310 gear steel which has been carburized to 1% carbon. The ausrolling process is an advanced thermomechanical processing technique used to ausform only the critical surface layer of gears and produce a hard, tough, fine-grained martensitic product. By eliminating the need for deformation of the entire bulk of the gear, ausrolling brings ausforming to a feasible and cost effective option for gears. The superior martensitic product formed by ausrolling has been shown to improve the rolling contact fatigue resistance of 9310 gear steel and could also improve the bending fatigue resistance of the gear steel. By improving the

rolling contact and bending fatigue resistance (both being significant causes of gear failure), industrial and defense gear applications could benefit from: improved gear life, smaller and/or lighter gears, and improved gear performance. This study compares the residual stress profile of a marquenched specimen with a moderately deformed ausrolled specimen and with a heavily deformed ausrolled specimen, in order to correlate the effects of residual stress with the improved fatigue properties of the gear steel.

### **NEUTRON DIFFRACTION RESIDUAL STRESS MEASUREMENT AT NIST**

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A neutron diffraction residual stress measurement program has been in place for several years at the National Institute of Standards and Technology (NIST). In this paper recent progress made within the scope of that program in two areas is described: 1) the development and initial performance of a new--semi-dedicated--stress measurement neutron diffractometer with a number of innovative features; 2) the application of the neutron technique to engineering-related problems. The latter includes studies of a variety of weldments: a spot weld and a v-notch weld in HSLA steel, and skip welds on tank-car steel (A515 grade 70); and the fabrication and characterization of a steel ring/plug residual stress reference specimen.

### **ON THE CALIBRATION OF MAGNETIC AND ULTRASONIC METHODS OF RESIDUAL STRESS MEASUREMENTS IN COLD ROLLED IRON- DISKS BY NEUTRON DIFFRACTION TECHNIQUE**

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Herold, Fraunhofer-Einrichtung IUW Chemnitz; & E. Schneider, J. Schreiber, & W.  
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Variation of internal stress states in cold rolled sheet metal can essentially influence the result of forming processes. Therefore it is important to control the forming process by a practicable in line testing method. For this purpose magnetic and ultrasonic nondestructive methods are available. However, it is necessary to calibrate these techniques. This paper describes a first step of such a calibration procedure making use of the neutron diffraction method. On the basis of the diffraction results an assessment of the magnetic and ultrasonic methods for the estimation of residual stress in the cold rolled iron-disks was made.

With the help of the high resolution Fourier diffractometer at the pulsed reactor IBR-2 in Dubna the strain tensor was measured at selected points of cold rolled iron-disks of 2.5 mm thickness. The complete strain tensor is determined from the measured reverse time of flight diffraction spectra for different orientations of the scattering vector. The

lattice spacing of the unstrained state was known from an annealed powder sample of the disk material. To obtain the change in the texture at different inspection points of the disk a parameterized texture model was included into Rietveld Refinement. On the basis of the diffraction results and the forming process outcome an assessment of the magnetic and ultrasonic methods was made. Reasonable measuring concepts for practical applications to forming processes with cold rolled sheet metal are discussed.

### **LOCALIZED STRESS MEASUREMENT OF ALUMINUM ALLOY WITH AN ACOUSTIC MICROSCOPE**

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With an acoustic microscope of a line-focused lens, stresses around a small hole have measured for an aluminum plate under tension. The residual stresses have also determined for that plate after shrink fit of a plug to the hole.

Combining the acoustoelastic law of the surface waves with velocity measurement of the wave, we can evaluate stresses near the surface of solid. The advent of an acoustic microscope, particularly the  $V(z)$  curve approach, enable us to measure precisely the velocity of leaky surface wave,  $V_{lsw}$  within very localized region, say a few hundreds micron. The acoustoelastic constants, namely the relative velocity change per unit stress, of common metals are of the order of  $10^{-5}/\text{MPa}$ , therefore, we should measure the relative velocity change up to  $10^{-4}$  for measuring stress within some 10MPa.

By modifying an acoustic microscope Olympus UH-3 and devising a digital signal processing for precise determination of the oscillating interval  $\Delta z$ , we succeeded to measure  $V_{lsw}$  within relative precision of  $10^{-4}$ .

The acoustoelastic constant of aluminum alloy 2017 has been determined by simple tension tests under the acoustic microscope. The value is about  $2 \times 10^{-5}/\text{MPa}$ . The material showed slight acoustic anisotropy. After making a hole within the identical specimen used for the above test, we measured  $V_{lsw}$  in orthogonal directions at several points around the hole under simple tension. Then a plug was force-fitted into the hole. Again the  $V_{lsw}$  was measured and the residual stresses were calculated with the acoustoelastic law. The measured stresses were compared with those obtained by FEM analysis. Generally, both results are in good correlation, however, the measured stress showed some variation due to insufficient numbers of crystallite with the focused area.

## **ULTRASONIC EVALUATION OF STRESS STATES IN RAILS**

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There is an increasing demand for nondestructive techniques to evaluate stress states in railroad rails. Papers have been published describing different methods and approaches using magnetic, magneto-elastic and ultrasonic techniques.

This paper summarizes the results of a study to optimize ultrasonic techniques for field applications.

Using rails from different manufacturers, the elastic and acousto-elastic constants as well as their temperature dependences are evaluated. The influence of the stresses along the length and the width of the rail are taken into account. The texture of the new and used rails has been investigated. Partially destructive techniques have been applied to analyze the real stress profiles and gradients in new and used rails. Based on these results, different ultrasonic techniques have been investigated with respect to the local resolution, accuracy, applicability and possibilities for the discrimination of local irregularities. EMAT-Transmitter-Receiver Units have been built and their temperature dependence has been measured.

Based on these investigations, two different ultrasonic techniques have been developed and tested for evaluating stresses in the new and used rails.

Experimental results of the in-field stress analysis as well as of the evaluation of thermal induced stresses are presented and discussed.

## **ACOUSTOELASTIC DETERMINATION OF STRESSES IN STEEL USING RAYLEIGH ULTRASONIC WAVES**

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The acoustoelastic effect, i.e. the stress dependence of the propagation velocity of ultrasonic waves in deformed elastic media, is of great interest as a nondestructive tool in the determination of applied and residual stresses. Rayleigh ultrasonic waves penetrate the material to a depth of approximately one wavelength. for this reason they have been chosen with the purpose of measuring sub-surface stress fields which are present in pieces after cold-working or heat treatments.

A device to make the measurements has been constructed with one transmitting and two receiving probes at fixed distance coupled to the material surface by means of springs. The probes are commercial piezoelectric transducers. The surface wave is detected by the said two receivers through two steel elements of different shapes (wedges, cylinders,

cones). The advantages of using one shape rather than another are illustrated. Two signals from the receiving transducers are fed to a digital oscilloscope and are mathematically correlated to determine their phase shift, i.e. the wave time of flight between the two transducers. Different correlation methods are examined.

The system is calibrated by measuring the acoustoelastic effect in a beam subjected to four point bending with known loads. The results and the experimental errors are shown and examined.

### **THE EDDY CURRENT TECHNIQUE FOR DETERMINING RESIDUAL STRESSES IN STEELS**

M. Blaszkiewicz & L. Albertin, Westinghouse Science and Technology Center, USA

Eddy Current (EC) testing has been successfully employed to determine the residual stress patterns in steels. Since any change in residual stress in ferromagnetic materials produces changes in the electrical and magnetic properties of the material, EC can be used to detect these changes in residual stress as changes in the impedance of the EC results. In some uses of steel parts, it is desirable for certain residual stresses to be present. By employing an NDE technique (EC) that is incorporated into the early stages of the manufacturing process, the steel parts which do not meet the required residual stress can be identified and discarded. In this way the service lifetimes can be improved dramatically. In our experiments excellent correlation was established between the magnitude of the stress as determined by the X-ray diffraction technique and the EC technique.

### **ANOTHER APPROACH TO ACOUSTOELASTICITY**

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A range of non-destructive techniques based on x-ray and neutron diffraction exists for determining the strain on a particular lattice plane of a bulk material. The physics of diffraction is well understood, and both techniques are capable of good spatial resolution, although x-ray is limited to measuring the stresses near the surface. Neutrons samples a larger volume, but require the presence of a high flux reactor. So there is a need for a cheap, easy and accurate technique, like elastooptics, which will provide a quantitative measure of the stress distribution in opaque solid materials. The ability to nondestructively map internal stresses would be particularly useful for critical components such as engine parts that may fail catastrophically if internal stresses are too high or for a variety of layered materials that may delaminate under stress. The acoustic microscope technique relies on computer analysis of acoustic waves that are directed through a material and reflected back to an acoustic microscope lens. Stressed areas of the material change the amplitude of the reflected polarized waves compared with unstressed areas. Previous applications of acoustoelasticity were focused mainly on the effect of stress-induced velocity changes of the acoustic waves. The maximum change in the ultrasonic wave velocity caused by the

presence of a stress is usually less than 1% of the velocity measured at zero stresses.

During the past two years, we have developed a new ultrasonic technique for the evaluation of stress distributions based on very accurate monitoring of the amplitude of the ultrasonic wave. Our earlier papers established how the amplitude height is affected by the polarization realignment due to stress and resulting interference of polarized acoustic waves at water-solid or air-solid interfaces in acoustic microscopy can be used for a quantitative measure of stress distributions for certain configurations of principal stresses. In contrast, acoustoelastic methods based on velocity changes are usually limited to evaluation of uniaxial or a plane state of stress.

The general theory is developed in cooperation with J.R. Willis from Cambridge University for the amplitude dependence of arbitrary shear waves propagating in arbitrary directions relative to existing principal stress directions. The theory was formulated for materials which, in the absence of stress, are assumed to be elastically isotropic.

The overall goal of the work is to develop a practical test that will allow manufacturers to remove not only products with cracks and other deficiencies from the production line but also those that are likely to fail in the future due to high internal stress.

## **RECENT DEVELOPMENTS IN NON-DESTRUCTIVE CHARACTERIZATION OF AEROSOLS BY SYNCHROTRON RADIATION**

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Aerosol pyrolysis has become a powerful technique of processing fine powders from solutions with improved physical and chemical characteristics. In recent years, new spectroscopies have been developed for the investigation at the micrometer scale of spray particles. *In-situ* spectroscopy analysis, as on line X-ray Absorption, is the best structural tool at the atomic level giving a description of the precursor transformation during powder production. We have shown that high purity, chemical homogeneity, particle size and sphericity which are requested for improving the powder quality, are related to the structural information during the synthesis from the liquid phase.

The principal advantage of this spectrometry is that it is particularly suitable for the study of disordered, glassy or vitreous materials. X-ray Absorption Spectroscopy is based on the determination of the attenuation of an X-ray beam produced by a synchrotron source in a condensed medium. The atomic distribution of a selected atom is obtained by Fourier transforming the XAS signal, as a function of incident photon wave number. The oscillations depend essentially on the number, the distance, the disorder and the type of the nearest neighbour of the excited atom.

The experimental set up used for *in-situ* X-ray absorption measurements of aerosols is composed of an ultrasonic nebulizer coupled with a detection chamber. We note two

important advantages of the ultrasonic nebulizer: first the aerosol is practically monodispersed and secondly both aerosol production rate and carrier gas flow rate can be independently varied. The micrometer-sized spheres are circulated in the X-ray irradiation cell for XAS experiments. The spray chamber is designed to promote a good mixing and transport of the aerosol towards the analysis cell where the droplets are irradiated. Spectroscopic measurements were performed at the synchrotron radiation facilities of LURE (Orsay, FRANCE).

Structural study by X-ray Absorption Spectroscopy has shown that the application of the ultrasonic nebulization technique to the synthesis of ceramic leads to materials composed of homogeneous and narrow size distributed powders of zirconia with the required particle morphology and size distribution suitable for sintering. The different stages of the application of heat treatment appears to control the alteration of particle morphology. Absorption experiments have evidenced that the crystallites growth from the amorphous phase is strongly related to the local structural development around the cations. The nebulization process which produces spherical particles highly qualified for sintering, should have important implications in the future for ceramic powder production as shown by the information derived by X-ray Absorption Spectroscopy about the evolution of the cation environment during the spray pyrolysis of precursors.

### **DETERMINATION OF SINGLE CRYSTAL ORIENTATION FROM OSCILLATORY BRAGG PEAK POSITION**

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Knowledge of the orientation of a single crystal is essential in many engineering applications. This work describes the theoretical background of an x-ray diffraction technique for the precise determination of the orientation of a single crystal. The analytical solution of a set of nonlinear equations describing the crystal orientation is a sine or cosine function, which is determined by a least squares fit of the experimental x-ray diffraction data. A fixed detector method is based on omega scans (omega is a rotation angle around an axis laying in the plane of the crystal surface and perpendicular to the diffraction circle) at various phi positions (phi is a rotation angle around the axis perpendicular to the crystal surface). The theoretical prediction of the oscillatory Bragg peak position in those omega scans was confirmed experimentally. The technique was used to determine the crystal orientation of a silicon wafer whose (100) plane was inclined at a small angle to the wafer surface. The experiments were carried out on a four axis goniometer using CuK-alpha radiation and (400) Si reflection.



## **NONDESTRUCTIVE MEASUREMENT OF GRAIN SIZE IN STEEL PLATE BY USING X-RAY DIFFRACTION**

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Microstructures of steel products such as grain size and texture are controlled to improve their quality in steel industries. The development of in-process measurement techniques to characterize the microstructures is strongly demanded. The Committee on Sensor for Microstructure was organized in Japan to study nondestructive methods for measuring the grain size. Round robin tests were carried out using following three methods in the committee: X-ray diffraction, ultrasonic attenuation and magnetic coercive force. The result of the round robin test using the X-ray diffraction method will be reported in this paper.

Following two relations are investigated in the round robin test: one is the relation between the grain size and the number of diffraction spots which appear in the Debye-Scherrer Ring and another is that between the grain size and the statistical variation in measured integrated X-ray intensity during a specimen is rotating. Many specimens with different grain size, texture, secondary phase (pearlite, martensite) and distribution of the grain size are prepared for the round robin test. The results of the investigation are as follows:

- (1) There is a good relation between the grain size and each measured values obtained in the round robin test unless disturbance factors such as texture and secondary phase exist.
- (2) Texture, secondary phase and distribution of grain size have great influences on the relation between the grain size and the measured value of the statistical variation.
- (3) The influence of the texture can be smaller if a suitable diffraction plan is chosen.
- (4) There is a possibility to measure the grain size with accuracy of  $\pm 1$  in ASTM class by the X-ray diffraction method.

## **HYDRIDING CHARACTERISTICS OF V-0.5 AT.%C ALLOY**

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Vanadium hydrides are potential thermal energy storage materials in heat pump applications. In this study, the hydriding properties of V-0.5at.%C ( $V_c$ ) alloy were investigated. Long term stability of the alloy was determined by hydrogen thermal cycling between the tetragonal  $V_cH$  and the cubic  $V_cH_2$  phases. Results showed increased

isotherm hysteresis as a function of number of cycles but without any significant changes in the desorption isotherm pressures. The isotherms were obtained after activation, 778, 1345, and 1633 cycles. X-ray diffraction results revealed that the microstrains,  $\langle \epsilon^2 \rangle^{1/2}$ , in the  $V_cH$  as well as in the  $V_cH_2$  phases decreased after cycling, attributed to the recovery processes. These microstrains were determined by using the Warren-Averbach method. Residual lattice microstrains were present even in the dehydrogenated  $\alpha$  phase obtained by heating the hydrides in a differential thermal analyzer. Thus, monitoring the pressures and the knowledge of lattice microstrains and long range strains during cycling reveals the condition of the alloy during long term operation of hydride heat pumps. Pre-straining this alloy also increased the hydriding hysteresis and decreased the microstrains, without any thermal cycling. Thus, lattice strains present prior to cycling are important. The effects of hydride cycling and cold work on the isotherm pressures, microstrains, long range strains and domain sizes in the  $V_c$  hydrides are presented.

### X-RAY DIFFRACTION CHARACTERIZATION OF THIN POLYCRYSTALLINE FILMS

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The physical and mechanical properties of thin films are often different from the properties of bulk material. Those properties are dictated by the film/substrate orientation relationship, crystal anisotropy and crystallographic texture of the film. X-ray diffraction texture analysis provides the information about preferential film growth and can be used for optimization of deposition parameters and prediction of properties of thin films.

An X-ray back reflection technique using the Bragg-Brentano geometry with experimental corrections for absorption and defocusing was used to study thin ceramic films deposited by combustion chemical vapor deposition (CCVD). The effects of deposition temperature and  $Y_2O_3$  content on the epitaxial relationships between  $Y_2O_3$ - $ZrO_2$  film and a (100) single crystal sapphire substrate, and film texture were studied. In the 2.5 mol%  $Y_2O_3$ - $ZrO_2$  film (tetragonal) deposited at 1200°C, {001}, {511} and {211} fiber textures were observed. As the deposition temperature increased to 1400°C, this texture was found to be sharper showing only {001} and {211} fiber textures. In the cubic 8 mol%  $Y_2O_3$ - $ZrO_2$  film deposited at 1200°C, strong {100} and {531} fiber textures were observed.

The film/substrate orientation relationships of  $YBa_2Cu_3O_x$  (YBCO) superconducting thin films deposited via CCVD on single crystal MgO and polycrystalline silver substrates were also studied. The as-deposited films on single crystal (100) MgO substrates showed strongly preferential growth with the basal plane parallel to the substrate surface (c-axis up growth). Texture analysis showed two in-plane alignment orientations of the film with respect to the substrate, with YBCO [100] and [110] aligned with the [100]

MgO substrate. YBCO films deposited on roll-textured polycrystalline silver at temperatures between 850-950°C displayed c-axis up growth indicating that the orientation of the polycrystalline substrate did not induce detectable in-plane preferential growth of the YBCO.

## PRINCIPLE AND PRACTICE OF MODIFIED PROPORTIONAL FACTOR METHOD IN XRF ANALYSIS

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This paper specially describes the conception, principle, derivation of mathematical expression and calculation method of modified proportional factor method. When the proportional factor method apply to analyze the gold jewelry, except correction of the spectral line intensity, the proportional factor also must be corrected.

Suppose that  $I_{iA}$  and  $I_{iB}$  present the measured intensities of analytical element  $i$  in standard and sample, respectively,  $I_{iA}^r$  and  $I_{iB}^r$  present their correction intensities, if  $C_{iA} = C_{iB}$ , shape and area of the sample are variables, then  $I_{iA}^r > I_{iB}^r$ ,  $I_{iB}^r > I_{iB}$  and  $I_{iA} > I_{iB}$ . If we assume  $\Delta I_{iA} = I_{iA}^r - I_{iA}$  and  $\Delta I_{iB} = I_{iB}^r - k_{iB} I_{iB}$ ,  $k_{iB}$  is a variable constant, if we change the  $k_{iB}$  continuously, we always can find a  $k_{iB}$  value to satisfy following condition:

$$\Delta I_{iA} = \Delta I_{iB} = I_{iB}^r - k_{iB} I_{iB} \text{ -----(1)}$$

Now we simplify equation (1) to  $\Delta I_i = I_i^r - k_i I_i$ , in addition, let  $\Delta I_i / I_i^r = D_i$  and  $\Delta I_j / I_j^r = D_j$ .

Experiments show that there are excellent linear relationship between  $\Delta I_i$  and  $k_i$ ,

$$\text{then let: } \Delta I_i = I_i^r - k_i I_i = b_i k_i + a_i \text{ -----(2)}$$

$$\Delta I_j = I_j^r - k_j I_j = b_j k_j + a_j \text{ -----(3)}$$

where  $b_i(b_j)$  and  $a_i(a_j)$  are the slopes and intercepts of the straight lines. From above equations we can derive following expressions:

$$\frac{I_i^r}{I_j^r} = \frac{I_i}{I_j} (1 - D_j) \left( 1 + \frac{b_i}{I_i} + \frac{a_i}{k_i I_i} \right) \text{ ----- (4)}$$

$$A_{ij} = \eta_i A'_{ij}, A_{ij} = I_i^r / I_j^r, A'_{ij} = I_i / I_j, \eta_i = (1 - D_j) \left( 1 + \frac{b_i}{I_i} + \frac{a_i}{k_i I_i} \right)$$

i.e.

this is to say, if we know  $b_i$ ,  $a_i$ ,  $k_i$  and  $D_j$ , we can calculate the analytical results of each element in jewelry. How to get these values? There are 1 figure, 12 tables and 22 equations to answer these questions in this paper.

### **X-RAY BACKSCATTER TOMOGRAPHY: NDT POTENTIAL AND LIMITATIONS**

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Although techniques for materials characterization with backscatter radiation have been known for a long time, only recently have such methods offered promise of practical NDT capability. This is the result of advances in detector technology coupled with the development of low cost computer processing power. One system making use of these developments is the ComScan, an x ray backscatter tomography system developed by Philips. At NRL we have had considerable success with the ComScan in developing a one-sided NDT procedure for Navy sonar domes. We have had the opportunity to apply the instrument to several other NDT problems with mixed success. We find that the ComScan is often applicable, but it is not always the best approach for a particular NDT problem.

### **A X-RAY SENSITIVE CCD CAMERA SYSTEM AND ITS APPLICATION TO THE X-RAY DIFFRACTOMETRIC INVESTIGATION OF AREA SELECTIVE SEMICONDUCTOR EPITAXY**

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Based on the need of a two-dimensional position sensitive detector to investigate structured epitaxial layers, a specifically designed CCD camera system is used. Because of the intended use for diffractometric applications there are two important requirements: Firstly the camera should allow to resolve details of about 20  $\mu\text{m}$ , secondly it should be possible to detect single x-ray quanta of the used 8 keV radiation. The "indirect" method is utilized, where the x-ray quanta are first converted into visible light in a polycrystalline phosphor, and this light is amplified in a one-stage microchannel image intensifier. Because of the long absorption length of about ten to some tens of micrometers in phosphor materials, the selection of the phosphor thickness is always a trade-off between resolution and efficiency of the system. Furthermore the selection of the image intensifier is a trade-off between resolution and light intensity at the output of the intensifier. It could be shown that our system achieves about requirements.

By means of samples with SiGe/Si area selective epitaxy an application of this detector in high-resolution x-ray diffractometry to deliver a sample mapping will be presented. This mapping is produced "simultaneously" opposed to diffractometric mapping by scanning the sample or the x-ray beam. The system can be used to take x-ray topographs. Topographs are sensitive to strain on the scale of  $10^{-6}$ .

### **DETERMINATION OF THE ELASTIC BEHAVIOUR OF CARBON-REINFORCED CARBON MATERIALS BY USING LASER-ULTRASONICS AND THEORETICAL MODELLING**

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High-temperature resistant light-weight materials for construction are in the focus of worldwide research. One aim of this research is to improve mechanical, thermal and chemical properties by structural optimization of these materials. An important parameter for the construction engineer is the elasticity of the material characterized by the elastic constants which can be determined by measuring ultrasonic velocities. We report on the determination of elastic wave velocities in carbon-reinforced carbon material using laser-ultrasonics. This technique allows for ultrasonic measurements at high temperatures due to the contact free generation of ultrasound by short laser pulses and the interferometric detection of the resulting surface displacement. Hence, measurements can be carried out remotely.

Elastic constants have been determined in materials at high temperatures by evaluating the time-of-flight for longitudinal as well as for transverse wave modes. This works particularly well in isotropic materials. In anisotropic media, however, things become more complicate. Assuming that the inspected material is transverse isotropic, it can be described by five elastic constants, thus requiring the measurement of two longitudinal and three transverse velocities in three different directions of wave propagation.

Since the CFC-material shows high ultrasonic attenuation and strong scattering due to its laminar structure, distinction between and interpretation of the different peaks occurring in the detected ultrasonic signal becomes very difficult. Combining measurements carried out at room temperature for the identification of the various modes with the ones obtained at higher temperatures, it is possible to obtain the temperature dependent elastic constants. First results will be shown, comparing theoretical modelling and experiment as well as measurements of the sound velocity up to  $1400^{\circ}\text{C}$ .

## **ELASTIC MODULI MEASUREMENTS OF SiC REINFORCED ALUMINA CERAMICS AT HIGH TEMPERATURES USING LASER-ULTRASONICS**

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Laser-ultrasonics is a technique to remotely generate and detect acoustic waves using lasers. Being a truly remote technique (standoff distances of the order of tens of cm or more), it is especially well suited for ultrasonic measurements at high temperatures, where conventional ultrasonic methods are difficult. Additionally, laser ultrasonics allows the simultaneous measurement of shear and longitudinal acoustic wave velocities. These velocities were measured in SiC whiskers reinforced alumina samples of 58 to 99% of maximum theoretical mass density in the temperature range of 20°C to 1800°C. Knowing the mass density and thermal expansion behavior of the samples, the measured velocities are related to the average bulk elastic moduli. At room temperature, the elastic moduli vary linearly by one order of magnitude with mass density (porosity). At temperatures of up to 1300°C, the elastic moduli decrease linearly and independently of mass density (porosity) with temperature. Above 1300°C, the elastic moduli may either increase or decrease depending on the sample mass density. Some time-dependence measurements at high temperatures were obtained. However, the samples sublimated and the condensate eventually blocked the furnace's optical ports, thus limiting the time window to a few hours.

## **CHARACTERIZATION OF CREEP DAMAGE BY ABSORPTION MEASUREMENTS USING LASER ULTRASOUND**

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& J. Reszat & M. Paul, Fraunhofer-Institut für zerstörungsfreie  
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In recent research activities nondestructive evaluation of material damage due to creep, fatigue, etc. in components is gaining importance for life prediction and extension programs. Among these the assessment of creep damage in low alloy ferritic steels which are widely used in piping systems in fossil plants is one of the topics of interest. It has been reported that the dependence of ultrasonic velocity on creep damage in low alloy ferritic steels has shown encouraging results. Since creep damage has influence on the change in microstructure due to formation of cavities it was anticipated that creep damage should also affect ultrasonic absorption characteristics of the material. In order to find a dependence of absorption on creep damage a study has been performed on small specimens cut from standard creep specimens. These creep specimens which were exposed to graded, i.e. time-limited creep tests at different temperatures and loads were made from new as well as from service exposed material (1/2Cr1/2Mo1/4V and 1Cr1/2Mo). For the absorption measurements the reverberation technique was applied using laser generated ultrasound and heterodyne interferometer as ultrasonic receiver. It has been found that the absorption exhibits a good correlation with accumulated strain and decrease in density. In the paper

exhibits a good correlation with accumulated strain and decrease in density. In the paper the experimental arrangement, metrology, the experimental results, and also the feasibility of the technique to the field are discussed.

### **NONCONTACT ALTERNATIVES TO LASER DETECTION OF ULTRASONIC SIGNALS**

J.W. Wagner, D.A. Oursler, & T.W. Murray, Johns Hopkins University, USA

Laser ultrasonic technology has been well characterized and satisfactorily developed, at least for many laboratory uses. This noncontacting and remote ultrasonic transduction technology has been demonstrated in a range of materials characterization applications, especially for specimens at elevated temperatures or those of an extremely fragile nature such as thin films. However, the principal limiting factor of all-laser ultrasonic systems has been the relatively poor detectability of the laser receiver. While research continues to improve the detectability of laser interferometric systems, alternative detection schemes, including air-coupled piezoelectrics, microwave interferometry, and electromagnetic acoustic transducers (EMATs), have been considered. Hybrid noncontact ultrasonic systems using laser generation and one of these alternate methods for noncontact detection are being explored. Although none of the hybrid systems offer the same flexibility and advantages for remote ultrasonic inspection that would obtain from a highly sensitive all-laser system, each offers its own set of advantages including lower cost, and in most cases, greater convenience than an all-laser system.

### **NICKEL-BASE SUPERALLOYS CHARACTERIZED BY SLAM AFTER LONG TERM HEATING**

V.A.M. Luprano & G. Montagna, Pastis-CNRSM, Italy

Superalloys, used for blades and vanes in gas turbine and jet engine due to the exposure to a very high temperature, tend to suffer from the change in their microstructure in the course of the long term service. To prevent premature failure from thermal fatigue and loading stresses, it is essential to establish the structural integrity of each blade. The effects of the long term heating for up to 10,000 hours of Nickel-base superalloys (INCONEL 738) are studied by the change of the acoustic attenuation (investigated by SLAM) due to the varying of the mechanical and thermal fatigue properties and the microstructure (investigated by SEM and TEM).

The SLAM (Scanning Laser Acoustic Microscope) system uses a piezoelectric transducer to produce plane continuous ultrasonic waves at the frequencies of 10, 30, 100 MHz and a scanning laser beam to detect the amplitude of the ultrasonic waves after their propagation inside the sample. Absorption, scattering or reflection of the ultrasonic waves crossing the sample are due to the changes of the elastic properties and/or density inside the sample.

Aim of this work is to investigate the correlation between the well known changes in the microstructure (phase transformation, creation of carbide at the grain boundaries and so on) of the Inconel 738 and the change of the acoustic attenuation in order to check the embrittle of the structure by means of nondestructive testing. Using SLAM technique in fact it is possible to obtain important information not only on the presence of defects, but also about the change of the microstructure thanks to the use of acoustical image. A further goal could be the correlation between the acoustical images and the pulse echo signal to use for nondestructive testing in service.

### **LMM-1 LASER MICROANALYSER OF MATERIALS**

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The LMM-1 laser microanalyser realizes for the first time a patented device, which permits a quantitative analysis of the composition of both metals and materials of the non-metal group, including thin metal and dielectric coatings (from 0.2 to 2 micrometers).

A number of well-known west companies is actively developing various kinds of analytical equipment for the determination of the chemical element composition of substances and materials, but analysers manufactured by them are of the X-ray type and can be used only for the metal group of elements.

Analysers, which are based on the arc discharge principle, have a number of essential disadvantages: they require much material for sampling purposes, and this makes it impossible to analyze noble metals and finished articles, as well as complex geological samples.

The spark analysers employ low-level radiation sources and therefore can be used only for the investigation of metals and their alloys.

The proposed laser emission microanalyser embraces all the performance potentials of the above-indicated instruments. The principle of its operation is based on the interaction between a powerful strictly metered stabilized laser radiation and the substance of the investigated sample and on recording the composition of the emission plasma by an optical spectrometer, provided this process is controlled automatically and the energy parameters of the laser source are controlled by a computer.



This analyser has the following main parameters:

Analysed elements	from Li (No. 3) to U (No. 92)
Dynamic range of concentration measurements (depending on the number of analysed element)	( $10^{-3}$ - $10^{-2}$ to 100%)
Maximum concentration measurement error	1%
Measurement time	1 min

The essential advantages of the LMM-1 microanalyser as compared with the analogues are ensured by:

- the possibility of analysing a wider class of substances and materials (practically all the materials of the non-metal group: plastic materials, optical glass, medicines, geological samples, crystals and semiconducting materials for electronics)
- microsampling provided by the laser source; this makes it possible to analyse thin metal and dielectric films
- stabilization of the parameters of the laser source, which increases the reliability and precision of the concentration measurements to 1%.

The LMM-1 microanalyser is an ecologically pure and safe instrument.

### **MICROHARDNESS AND RAMAN SPECTROSCOPY FOR CHARACTERIZATION OF FULLERITE SINGLE CRYSTALS**

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Microhardness testing is the only nondestructive measuring method for plastomechanical parameters which also operates with specimens of very small volume, thickness and of arbitrary geometry, although in some cases an exact derivation of plastic flow stress is difficult. Recent equipments exhibit accuracies better than 1% by entirely automatic performance and also yield in-situ load/extension characteristics representing stress-strain dependences, examples of which we report here.

The Raman spectroscopy is a well proven method to investigate optical phonons and thus, the nature of atom bindings in certain solids. This method is particularly effective in systems with strong chromophoric character and high degree of degeneracy (like actual  $C_{60}$ -fullerite) where the spectra become rather simple thus allowing for investigation of symmetry breaking conditions.

In this paper, a combined investigation by microhardness and Raman spectroscopy on Fullerite single crystals is presented; in particular, their sensitivity to illumination by natural light is reported. When illuminating C<sub>60</sub>-crystals which were not exposed to air and light before, the value of Vickers microhardness H<sub>v</sub> markedly increases, while the pentagonal pinch mode A<sub>g</sub>(2) at 1468 cm<sup>-1</sup> of Raman spectrum dramatically decreases, although new lines with low frequencies correspondingly increase. On the contrary, no such effects are observed on oxygen stabilized C<sub>60</sub>-crystals.

The experimental results can be undoubtedly explained when a photoreaction between an excited C<sub>60</sub>-molecule and its neighbors is assumed which form a C<sub>60</sub>- dimer or oligomer. This process does not occur if oxygen is still present in the sample which is consistent with experimental results reported above.

## **VERY NEAR FIELD OPTICS, A FRONTIER TECHNOLOGY**

J.B. Goodell, Westinghouse Electric Corporation, USA

Optics of very near fields has achieved considerable importance as a result of rapid developments in frontier technologies. Among these are; large scale integration, micro-optics, data storage, and high resolution metrology, all major areas of modern optical science. In spite of this basic significance, Near Field Optics (NFO), until quite recently, has received little interest, except at a few research centers. Emphasis has been, almost always, on optical far fields. Thus, the importance of NFO to modern optical technology is little appreciated. An example of great practical significance is the extremely high spatial resolution ability of NFO, which easily exceeds the limits of Fraunhofer optics by an order of magnitude. Further, edge-of-technology devices which involve sub-micron dimensions, such as micro optics, scanning microscopes, and the like, require NFO theory for correct analysis. Thus, it is essential to understand NFO as applied to the highly sophisticated, state of the art instruments and devices.

We have demonstrated the significance of NFO in several of the above areas. In metrology we designed and manufactured what we believe to be the first high resolution absolute optical encoder using NFO design theory. The successful design, manufacture and operation of this encoder has great practical significance, not only for metrology, but other state-of-the-art applications that benefit from ultra high optical resolution, and other NFO features.

This paper reviews some NFO principles, including contrasts with far optical fields. Emphasis is on high spatial resolution potential. Discussion includes the encoder design, manufacture and operation. Potential applications are noted, such as surface measurements and scanning microscopy. Optical disc technology stands out as a possible prime beneficiary of the huge increase in data density that NFO enables.

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### **DELAYED CRACKING IN AUTOMOTIVE WINDSHIELDS**

S.T. Gulati, H.E. Hagy, & J.F. Bayne, Corning Incorporated, USA

The laminated safety windshield has served both automotive industry and the consumer for well over 30 years [1,2]. The invention of float process in early 1960's provided high quality soda-lime-silica sheet glass which, due to its low cost, found ready acceptance in automotive and architectural glazing. In view of its excellent surface quality and thermal temperability, its initial strength is respectably high ranging from 90 Mpa in the annealed state to 180 Mpa following tempering. In addition, its optical quality has surpassed the windshield requirements set by automakers for clear, undistorted, viewing by the driver.

The windshield manufacturing process, however, involves scoring of glass sheet, seaming or beveling of edges, contour bending, and lamination to PVB (poly-vinyl butyral) all of which require much handling and can introduce flaws notably at the edges. The bending step produces significant but controllable residual stresses due to the short thermal processing schedule. Edges are forced into beneficial compression, but adjacent regions can experience excessive membrane tension if thermal gradients are large. When the bent glass plies are laminated to a thin interlayer of PVB, significant bending stresses are introduced in high curvature regions due to small deviations in contour parallelism of the two plies. Although the edge compression provides each glass ply the much needed strength to withstand handling stresses, the combined tension from membrane and bending stresses in the adjacent region can result in delayed cracking of windshield due to poor stress corrosion resistance of soda-lime-silica glass. The magnitude of these stresses must be controlled to below the threshold value to prevent delayed cracking initiated by surface flaws induced by stone impact and/or abrasion by hard particles.

This paper will describe the use of Friedel polarimeter for measuring membrane stresses in each of the glass plies as well as in laminated windshield as function of manufacturing process, ply thickness and peripheral position. An abrasion test in 100%

RH helps verify the measured stress by recording the frequency and failure time for delayed cracking [3]. Methods to reduce tensile stresses, and hence delayed cracking, will be proposed. Fractography of windshields which exhibited delayed cracking in the field will be used to estimate the tensile stress and compare it to that obtained photoelastically. The abrasion test has an excellent potential for quality control test and should prove valuable to automakers worldwide.

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### **HIGH-BANDWIDTH, SELF-COMPENSATING, LASER-BASED ULTRASOUND DETECTOR USING PHOTO-INDUCED EMF IN GaAs**

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There is a need in the manufacturing sector to rapidly and nondestructively inspect components under harsh in-factory and field-testing conditions. Laser ultrasonics offers a technique for inspection without direct contacting, resulting in a nondestructive, remote diagnostic. Conventional interferometric and heterodyne receivers are unable to compensate for workpiece vibrations and/or wavefront distortions, relative platform motion, and local turbulence. Fabry-Perot-based systems have limited application due to complexity and cost. Compensated interferometers employing wavefront-matching or phase conjugation typically lack the speed-of-response to function in the factory. We describe a simple, inexpensive, adaptive photodetector based on the nonsteady-state photo-induced emf effect, which generates a time-varying output current in response to lateral motion of an incident optical pattern. The optical pattern stems from the interference of a probe-laser beam, scattered by a workpiece undergoing inspection, with a reference plane wave. Using a single crystal of GaAs:Cr as the detector element and a cw 532 nm laser as the source (with about 12 mW/cm<sup>2</sup> incident on the crystal), we demonstrated a sensitivity of 5 mrad phase shift (corresponding to about 1 nm of surface displacement) at 30 MHz, with a unity signal-to-noise ratio. We integrated the sensor into a prototype laser ultrasonic inspection system to evaluate its performance in measuring laser-generated ultrasonic pulses. The transmitter was a Q-switched 1.06  $\mu$ m Nd:YAG laser. The photo-emf sensor simply required a battery-powered low-impedance amplifier for processing, whereas a typical heterodyne system required an expensive phase-locked-loop post-processor to track low-frequency vibrations. The adaptive photodetector functioned well under dynamic conditions by gauging the thickness of a wobbling metallic plate, moving at speeds in excess of 1 m/sec. By contrast, the optical heterodyne system lost lock with even minor perturbations. The bandwidth, sensitivity and signal-to-noise of the adaptive photodetector

compare favorably with ideal interferometric receivers, and may be suitable for on-line industrial applications.

### **LOCK-IN VIBROTHERMOGRAPHY APPLIED FOR NDT OF POLYMER MATERIALS**

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and Kunststoffkunde, Germany

We present nondestructive testing method used on lock-in thermography with mechanical heat excitation. Stresses are generated in the sample by vibrating it either by a mechanical shaker or by an ultrasonic sender. The strong acoustical damping in the sample causes the mechanical energy to be converted to thermal energy. The defects in the sample act as regions of stronger damping resulting in higher temperature generation. Because of the change of the thermal properties, the defects affect also the heat conduction in the sample. Both phenomena result in thermal anomalies due to the defects, which can be detected with an infrared camera, for example.

In lock-in thermography, the high-frequency vibration is amplitude modulated with a low frequency. Magnitude and phase of the sample temperature in respect to the modulation are measured, in contrast to conventional thermography where only the dc-temperature is measured. This increases the reliability of the defect detection and, for example, the adverse effect of the inhomogeneous surface emissivity can be overcome. The use of high vibration frequencies gives good thermal signal already at low stress levels, helping thus to keep the testing truly nondestructive. Measurement results of CFRP and GFRP composites and different polymers with impact damages, delamination, and inclusion will be presented, as well as application examples for the evaluation of paint thicknesses on plastic and the quality of bonding.

### **PHOTOTHERMAL INVESTIGATION OF SILICON WAFERS WITH DIAMOND-LIKE COATING**

J. Bodzenta, J. Mazur & R. Bukowski, Silesian Technical University, Poland

Diamond-like carbon (DLC) layers become very popular for their interesting physical properties. The DLC layers have very high hardness and heat conductivity. They are applied as coating layers on different materials. One of the most interesting applications of these layers is covering of semiconductor devices. The diamond-like layer protects a device mechanically and improves heat abstraction. The problem, that should be solved is elaboration of methods for testing of DLC layers. The aim of the research, which is carried out in our laboratory is to work out photothermal methods for qualitative and quantitative characterization of layer structures with DLC layers.

Photothermal testing methods are nondestructive and noncontact ones. They use propagation of thermal waves for evaluation of thermal properties of a sample. Another parameters of the sample, that affecting its thermal properties, can be evaluated, too. Penetration depth of thermal wave depends on its frequency. Therefore measuring methods using thermal waves are very useful for characterization of layered samples.

Measurements are carried out for silicon wafers with and without DLC coating. Photodeflection method for photothermal signal registration is used. The signal dependencies on measuring setup geometry and frequency are determined. There are distinct differences between dependencies measured for coated and non-coated samples. These differences are bigger for higher frequencies.

The investigation has preliminary character. Received results shows possibility of using photothermal methods for characterization of layered samples with DLC layer. Further theoretical and experimental researches will be conducted for elaboration of quantitative methods of measurements. The methods should give possibility of determination of DLC layer parameters (thickness, thermal diffusivity, etc.).

### **HIGH RESOLUTION PHOTOTHERMAL IMAGING OF METAL MATRIX COMPOSITE INTERFACE**

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& U. Netzelmann, M. Disque & M. Kröning, Fraunhofer Institute  
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Using photothermal measurements as a new NDE method opens an application field of increasing interest for material characterization. As this high resolution micro-NDE especially applies to the near surface range of materials, it should be able to characterize material interface considerably. In this paper, the interface of zinc alloy metal matrix composites (MMC) were investigated by the photothermal microscope. Experimental results showed that the clear image of the MMC interface and the variation of interfacial condition controlled by fabrication process parameters can be indicated at different measuring frequencies. Furthermore, theoretical model and simulation were studied to correlate the experimental works.

### **NONDESTRUCTIVE TESTING OF LACQUER COATINGS**

B. Bendjus, B. Koehler & Th. Vetterlein, EADQ, Germany

Lacquer coatings on different materials like metals or synthetics play an important role for the protection of the substrates against mechanical and corrosion damage. The adhesion strength of the coating is an essential factor for the evaluation of corrosion beginning from the point of coating damage. The corrosion behavior of coatings is tested by long-time experiments under atmospheric conditions or in special climate chambers.

The long test duration and the extreme conditions as compared to reality on the one hand and the limited possibilities of testing coated synthetics on the other hand causes an increasing interest in test methods indicating the damage or delamination of lacquer coatings directly and nondestructively. The paper describes several methods investigated in our institute on different material combinations using model samples with defined damages as well as "real" samples. The main method--thermal wave microscopy--is based on the photothermal effect and uses a chopped laser beam lighting the sample surface. In this process a part of the beam is absorbed by the sample while another part is reflected. The time-varying absorption of energy cause a periodical heating of the sample. By the comparison of the intensity and/or phase shift of the thermal radiation with the intensity of the reflected light, sub-surface defects can be detected.

In thermography, the sample under test is heated from the rear producing so a temperature gradient and heat flow is through the same and the substrate-lacquer interface. Consequently, delaminations point out surface temperature variations which can be observed by a thermo camera. High frequency ultrasonic imaging can be used in a through transmission and in a pulse echo mode. If a delamination is connected with cavity formation, high ultrasonic reflection and a very low transmission result as compared to adjacent perfect areas. This leads to a good contrast of ultrasonic pictures. The different methods for lacquer substrate evaluation are compared and their special advantages and disadvantages for delamination detection are discussed.

## **STRUCTURAL AND MORPHOLOGICAL CHARACTERIZATION OF PARTICULATE CERAMIC MATERIALS BY INFRARED SPECTROSCOPY**

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Infrared spectroscopy is shown to be a powerful tool for the characterization of ceramic powders with controlled particle size and shape, which are highly desirable for ceramic processing . Thus, it gives information not only on the composition of the powders but also on some structural aspects such as the orientation of the crystallographic axes in the particles (1), and on the shape and state of aggregation of the latter (1-3). This will be illustrated by using several ceramic powdered oxides with uniform particle shape and different ionic character ( $\text{TiO}_2$ ,  $\alpha\text{-Fe}_2\text{O}_3$ ,  $\text{SnO}_2$ ,  $\text{SiO}_2$ ). The infrared spectra are analyzed with the Theory of the Average Dielectric Constant, which takes into account the particle morphology through a shape factor ( $g$ ) related to the particle axial ratio, and the aggregation state, through a filling factor ( $f$ ) defined as the volume fraction occupied by the solid in the infrared pellet.

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## EVOLVED HEAT AS A FATIGUE CHARACTERIZING PARAMETER

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The study of fatigue and fracture has in the past relied predominantly on knowledge of the stress/strain distribution at the crack tip to characterize the 'criticality' of a crack. The stress intensity factor (K) approach is probably the best example of this. There are however many cases in which it is difficult, and indeed in some inadequate, to implement the K approach. Crack closure is one phenomenon known to significantly influence stress intensity factor type characterizations. Although in theory it can be suitably accommodated if closure stresses are well estimated, i.e. the concept of  $K_{\text{eff}}$ , there are many doubts about the ability to estimate closure stresses. Large scale plasticity is perhaps the prime example where the K approach fails. It was acknowledgement of the presence of plasticity at the crack tip that prompted a revision of theory which led to the development of the J-integral, a somewhat more appropriate parameter with which to characterize the state of a crack when appreciable plasticity is present. Unfortunately the J-integral also has shortfalls.

Our research has explored the use of the heat evolved at the crack tip as a characterizing parameter. One of the most significant advantages of this parameter is that phenomena such as plasticity and crack closure are accounted for. The reason why it has not been explored as a practical characterization parameter is probably due to experimental limitations. One of the requirements is to adequately map, both spatially and temporally, the temperature response in the vicinity of the crack tip. This has only recently been made possible by progress in infrared camera technology.

This paper will present an experimental/numerical technique for the determination of the evolved heat at a fatigue crack tip. A state-of-the-art infrared camera is used to map the temperature distribution in the vicinity of a crack tip. The temperature response thus gained is then used in an inverse estimation algorithm to determine the heat source over the mapped region.



Experimental results to date show that the crack propagation rate is extremely well correlated with the evolved heat, better so than with &K. This was not surprising as the evolved heat implicitly accounts for both plasticity and crack closure, in contrast to &K.

Given the results so far and the practical efficiency of the method, it is envisaged that the system could be used as an NDE tool for the in-situ characterization of the 'criticality' of a fatigue crack.

### **CHARACTERIZATION OF THERMAL DETERIORATION OF STAINLESS STEEL WITH ULTRASONIC VELOCITIES AND BACKSCATTERING NOISE**

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The deterioration of SUS316L steel under thermal and mechanical loading has been evaluated with the change in the ultrasonic velocities and backscattering intensity. The tested samples were cut out from a distributing unit, which was used for 27 months, of a blast furnace. Under the loadings, the microstructure changes with time, namely, chromium carbide particles precipitate within grains as well as grain boundary, and microvoids are formed. Thus the mechanical properties change with the increase in time.

To characterize nondestructively these microstructural change, we measured the ultrasonic velocities as well as backscattering noise intensity.

The longitudinal velocity increased about 20m/s or 0.3% in rolled plane, but 40-6m/s or 0.7-1% in out-of-plane direction. Transverse velocity also increased 40m/s or 1.3% at maximum. These are caused by precipitation of very stiff chromium carbides.

The backscattering noise intensity was also measured for each face of the cubic samples with longitudinal as well as transverse waves. The backscattered noise intensity on time domain, namely the square of the amplitude, were transformed into the frequency domain. The area under power spectrum is taken a noise parameter. The ratio of this value to that of virgin specimen is on the propagating direction of ultrasonic waves. The optical and SEM micrographs depicted the cylindrical cavities corresponding to the directional dependence of the backscattered noise. This backscattered noise measurement is particularly useful for large structures, because the measurement is possible in-service condition

## **NONDESTRUCTIVE INSPECTION OF TURBINE BLADES WITH LOCKIN THERMOGRAPHY**

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Lockin thermography based on the principle of photothermal radiometry has proven to be a fast and sensitive technique for NDT applications. The principle of the method is to measure thermal wave phase shifts induced by local variations of material properties. Even at low modulation frequencies (e.g. 0.03 Hz) the measurement time is only about 3 minutes.

From a practical point of view, it is highly desirable to characterize the mechanical properties of heat resistant coating for turbine blades such as density, porosity, and hardness variations.

We will present results obtained on some relevant problems related with turbine blades:

- monitoring of wall thickness variations,
- wall thickness variations of a wax model (which is constructed for cast) can also be detected, this is of the most interest for quality control in the early stage,
- correlation of phase angle with mechanical properties,
- defect detection in metal under a ceramic layer or under thin metal layer.

## **PARAMETER ESTIMATION IN PHOTOTHERMAL MEASUREMENTS WITH PHOTODEFLECTION DETECTION**

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Recently fast progress of nondestructive testing methods using thermal waves has been observed. These methods base on periodical sample heating by modulated light beam. A nonstationary temperature field (described as thermal wave) is generated in the sample. One of the most popular photothermal measurement technique is the method, in which "mirage" effect for signal detection is used. This detection technique is known as photodeflection detection. A probe light beam is deflected by a thermal lens - non-equilibrium distribution of refractive index caused by nonequilibrium thermal field. The photodeflection detection gives possibility of non-contact measurements.

An amplitude and a phase of probe beam deflection is measured by four-segment photodiode. According to the elaborated theoretical description the measured signal depends on many parameters [1]. In the simplest case, when the probe beam is a Gaussian one with a narrowing in a region of thermal lens and the sample is homogenous, isotropic with high light absorption coefficient the most important parameters are: power beam radius on the sample surface  $a$ , Gaussian radius of probe beam  $R$ , distance between the probe beam and the sample surface  $h$ , thermal conductivity coefficients of the sample  $\kappa_s$  and gas above it  $\kappa_g$ , thermal diffusivities of the sample  $\beta_s$  and the gas  $\beta_g$ , and modulation frequency  $\omega$ . In addition, in measured signal analysis the parameters of electronic measuring chain (amplification  $C$ ) and noise  $S$  should be taken into account.

Among parameters mentioned above only  $\omega$  can be easily determined. The rest of them are either unknown at all (e.g.  $C$ ,  $S$ ,  $\kappa_s$ ,  $\beta_s$ ) or only approximately known (e.g.  $a$ ,  $R$ ,  $h$ ). So, for proper analysis of measured data all of these parameters should be estimated. It is especially necessary when the measuring setup is tested.

The procedure for estimation of all mentioned parameters is worked out. For the estimation the least square method for complex function with linear anamorphosis and iteration methods are used. Because of nonlinear signal dependence on most parameters a minimum of a fitting function is numerically searched. As a result of the fitting procedure estimator values of all or selected parameters with appropriate uncertainties are obtained.

- [1] J. Bodzenta, R. Bukowski, Z. Kleszczewski, J. Mazur, *The modified theory of photodeflection detection applied in photothermal measurements*, prepared for publication in J. Appl. Phys.

## **THERMOACOUSTIC VIBROMETRY FOR IN-SITU MONITORING OF PROCESSES**

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The quality of the manufacturing process substantially determines the quality of the materials produced. Therefore one is interested in testing methods that are sensitive to the key parameters of the production process.

The details of the sintering process are hard to quantify since in-situ measurements are very difficult due to the high temperatures involved and the limited mechanical access to the samples. We developed a technique that allows a remote vibration analysis by means of optical excitation and detection.

Absorption of an intensity modulated laser beam induces periodical thermal expansion and consequent vibrations of the sample. Variation of the modulation frequency, interferometric detection and lock-in-analysis of the signal allow to follow the resonance curve and its width through the process.

This method can be used for the other demanding applications. The drying process of paint and the curing of epoxy resin demonstrate the flexibility of this approach.

#### **A NON DESTRUCTIVE TECHNIQUE, THERMAL WAVE IMAGING TO CHARACTERIZE THE ELECTROMIGRATION OF Al ALLOY**

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In submicron integrated circuits on Si, aluminium and its alloys are employed in the different metallisation levels. A critical issue in the performance of the final product life, is the resistance to the electromigration of the Al alloy. It has been found that electromigration is sensitive to the structure of the alloy.

In this study we have used a non destructive technique, Thermal wave Imaging, to characterize the microstructure of the Al metallisation films in terms of grain morphology and grain size. Further, we have investigated the metal sensitivity to stress and correlated the life time dispersions after classical electromigration in a furnace with process parameters typically used in microcircuit multilevel AlSiCu (1%/0,5%) metallizations.

In the Thermal Wave Imager, it is possible to create two different types of the image. In one image, a modulated (1 MHz) pump beam (Ar laser 488nm) is focussed with a 0,5 mm spot size on the sample. The modulated light is partly absorbed, generating a heat wave. A continuous wave HeNe laser (633nm) probes the reflectivity modulation &R induced by the temperature variations. An X-Y stage allows the signal to be imaged with submicrometer precision. In the second type of image, the orientation of the reflected light is detected and displayed in terms of the X and Y displacements of the specimen. With the Thermal Wave Imager it is also possible to create a sub-surface image. After acquisition of an image of either type, the Image Analysis Software allows a statistical evaluation of the AlCu grains to be obtained.

#### **RESIDUAL STRESS DISTRIBUTIONS IN THE RIM OF A STEAM TURBINE DISK USING THE L<sub>CR</sub> ULTRASONIC TECHNIQUE**

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Using critically refracted longitudinal (L<sub>CR</sub>) waves, the circumferential residual stress field has been studied in the rim of a pearlitic steel disk used for steam turbines. With the disk intact, maximum stress variations of 21.2 MPa from the mean were found on

the inlet face. Two sections, approximately 250 mm in length, were removed from the rim. Before and after travel-times collected from these cut sections indicate an initial stress differential between the inlet and outlet sides. For one section, estimated stress values before cutting are +176 MPa and -80.3 MPa for the inlet and outlet faces, respectively. For section two, similar values are -16.2 MPa and 30.9 MPa. One section which was thermally stress relieved showed a stress reduction of +275 MPa and -73.8 MPa on each side as a result of the stress relief. During the course of the work, the effectiveness of the  $L_{CR}$  technique was demonstrated by mechanically applying stress to the disk and measuring the travel-time change. Further, stresses at the various stages were monitored both with strain gauges and x-ray diffraction. While there is some disagreement in the quantitative values obtained with the three techniques, the differences are felt to be due to the different depths being interrogated by each method. The 2 MHz  $L_{CR}$  waves cover a layer approximately 3 mm below the surface. The other two methods are surface measurements. Thus, the  $L_{CR}$  data, are affected by the stress gradient and not just the surface stress.

## **NONDESTRUCTIVE EVALUATION OF NONCONDUCTIVE CYLINDRICAL NOZZLE IN PULSEWISE EXCITED QUASI- STATIONARY ELECTRIC FIELD**

Y. Bulbik, Siberian Aerospace Academy, Russia

Electric space propulsors are known to be effective in some space apparatuses either for their manoeuvring on the Earth-orbital neighborhood or for their transfer to higher orbits. The service time limiting main component of electric space propulsor as so-called closed-drift thruster (CDT) is the discharge ceramic chamber, or more simply the nozzle.

The CDT long-term prediction may be connected with nondestructive evaluation (NDE) of ceramic nozzle properties such as density, submicron structure of pores, chemical bond energy, electrical behavior, etc., especially through highlighting local electrophysical parameters without sampling.

Physically, the NDE technique at issue is based on a quasi-stationary electric field flux variation measurement by an electric charge transducer (ECT) placed on the ceramic nozzle wall external surface at the different temperature gradient conditions generated by an additional test heating from the opposite wall side. The ECT transference modelling problem is perceived here as two consecutive task. The first one deals with calculation of the electric field distribution in the ceramic nozzle wall at the zeroth temperature gradient and the second is based on an integral equation modified technique for an account of some material electric property gradient induced by the test heating. The required solution is reduced to truncated approximation by an interpolating polynomial of the electric potential distribution in an inhomogeneous medium with a five-initial partial derivatives on the angular and radial spatial variables in the circular cylindrical coordinates.

## **NONDESTRUCTIVE EVALUATION OF MATERIAL PARAMETERS USING NEURAL NETWORKS**

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For lifetime analysis the characterization of microstructure states and the determination of mechanical and technological parameters for different steel grades (20 MnMoNi 5 5, 22 NiMoCr 3 7, X20 CrMoV 12 1, 10 MnMoNi 5 5, 17 MnMoV 6 4, 15 MnCuMoNb 5 and WSTE) with nondestructive methods is an important objective. Toughness sensitive quantities must be employed, which can value changes of the dislocation density, or residual microstresses and precipitation states. Several micromagnetic measuring parameters derived from Barkhausen noise, incremental permeability and longitudinal magnetostriction were investigated. To determine tensile strength, yield strength, hardness and other parameters, it is necessary to combine several nondestructive measuring quantities in a multiparameter evaluation approach. This approach approximates an inverse function modelling the relation between measuring values and material parameters. It was realized using neural networks of the backpropagation type.

The approach was successful in separating different microstructure states for different material parameters. The best results were achieved using 14 different measuring values. In this case neural nets are superior compared with conventional numerical regression algorithms. But even in cases of direct (1 to 1) relation and combinations of up to 4 measuring values the neural nets achieved better results. In addition neural nets proved to be capable of supporting the optimization of NDE-technique by objectively evaluating the significance of nondestructive parameters, both as single values and as combinations of them. As a spin-off various other projects related to material science and based on the same ideas were launched. In future the work will be directed towards the usage of selforganizing maps as neural nets to improve the degree of generality of the method described.

## **NONDESTRUCTIVE THICKNESS DETERMINATION OF METALLIC COATINGS USING ULTRASONIC LEAKY RAYLEIGH WAVES**

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This paper presents a non destructive method based on the measurement of the ultrasonic Leaky Rayleigh waves characteristics (velocity and attenuation) for the thickness measurement of Vacuum Plasma Sprayed (VPS) NiCoCrAlY coatings deposited on AISI316L and Ni-based substrates which are used to protect gas turbine blades against high temperature corrosion and erosion phenomena.

The ultrasonic technique used is based on the measurement of an acoustic signature (the so called  $v(z)$ -curve) of the coated specimen. Two highly-damped flat transducers (one working as a transmitter, the other as a receiver) are translated along a  $z$ -axis, perpendicular to the sample surface. The sample is immersed in water. For each height  $z$  of the transducer, the received signal is digitized and Fourier transformed. Thus, for each frequency, complex  $v(z)$ -curves are obtained.

At a given frequency, when the thickness is low, the wave is entirely contained in the substrate and the attenuation is maximum because it is not only due to the radiation in water but also to the retrodiffusion on the grains. For the same frequency, when the thickness is very large, the wave is entirely contained in the coating material and the attenuation is now mainly due to the radiation in water because the finer grain structure of the layer gives less retrodiffusion. A similar reasoning can also be applied to the wave velocity. The variations of the received signal amplitude and phase are depending on the frequency, the coating thickness and the Rayleigh Wave (RW) velocity in the substrate and in the coating materials. It is also dependent on the shape of the beam. By choosing the dimensions of the beam appropriately, the height  $z$  and the frequency, the variations of the amplitude and of the phase allow the measurement of the thickness.

The method was applied to different substrates coated by a NiCoCrAlY layer with the thickness ranging from 60 to 330  $\mu\text{m}$ . The "ultrasonic" layer thickness was then successfully correlated to the one optically measured on cross-section micrographs.

Two cases were observed. The first one where the ratio between the RW velocities in the coating and in the substrate materials is  $\sim 0.85$ , allowing the phase analysis of the  $v(z)$ -curve to be well adapted. The second one where the ratio is larger ( $\sim 0.95$ ) so that the amplitude analysis of the  $v(z)$ -curve has to be performed, giving however a lower accuracy on the thickness. This work has been performed in the frame of a contract with ENEL spa (Italy).

## **NONLINEAR ULTRASONICS FOR MATERIALS CHARACTERIZATION**

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Nonlinear effects in elastic wave propagation are far more sensitive than linear properties, such as sound speed, to microstructure that is small in comparison with a wavelength. For example, microinhomogeneities associated with cracks, grains, and fibers can increase the effective nonlinearity of a material by several orders of magnitude, in comparison with the nonlinearity of homogeneous materials [L. A. Ostrovsky, J. Acoust. Soc. Am. **90**, 3332 (1991).] The three principal modes of elastic wave propagation--compression, shear, and surface--respond in significantly different ways to nonlinearity. Compression waves resemble sound waves in fluids, exhibiting quadratic nonlinearity that is independent of frequency. Plane shear waves exhibit cubic nonlinearity. Although surface wave nonlinearity is quadratic, it depends strongly on frequency. We shall review

briefly several methods of nonlinear nondestructive testing based on these different modes of propagation. In one investigation, an intense beam of ultrasound in water is incident on a thick plate, and harmonic generation due to nonlinear interaction of the compression and shear waves within the plate is studied. Pulse distortion in nonlinear Rayleigh waves excited by laser irradiation is considered. Finally, the effect of plate thickness on harmonic generation in nonlinear Lamb waves is taken into account. Theoretical models have been developed for each of these cases, and experiments are in progress. [Work supported by the National Science Foundation, the Office of Naval Research, and the Schlumberger Foundation.]

## **REVIEW OF INSPECTION QUALIFICATION PROGRAMME AND RRT RESULTS**

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The first phase of the inspection qualification programme conducted at NRI, Division of Integrity and Materials focused on personnel performance demonstration. The performed Round Robin Test (RRT) - blind test blocks trials - was performed on five specimens simulating critical primary circuit components of WWER 440 (V-213) and WWER 1000 (V-320) type NPPs. The specimens manufactured in Russia with implanted flaws simulating reactor pressure vessel (RPV) circumferential welds, clad/base interface and primary circuit pipes were inspected by manual and automated ultrasonic testing (UT) techniques and procedures in the conformity with independent auditor recommendations. These blind test trials were carried out by seven teams from four countries (U.K., Hungary, Slovak Republic and Czech Republic) in 1993-1994. It was shown that the inspection results assessed according to PISC statistical characteristics like DDP, DDF, CRP etc. correspond in detail with the PISC-II and PISC-III conclusions especially from the point of the effect of human factor in ultrasonic examinations. The specimens and implanted flaws specifications including UT inspection teams results were assessed and comprised with the ASME Code Section XI Appendix VIII requirements. RRT results were verified by radiographic examinations of the specimens.

The present phase of the inspection qualification ion programme is concentrated on performance demonstration capabilities of both ultrasonic testing equipment, ultrasonic techniques and in-service inspection (ISI) procedures used or intended for inspections of both the WWER 440 and WWER 1000 critical primary circuit components. This phase based on the information gathered on current inspection rules and evaluation of results relating to manufacture, pre-service and in-service inspection and applicable procedures used for inspection of critical primary circuit components includes:

- Implants definitions and flaw specifications typical for WWER NPPs including their manufacture and insert methods with the emphasis on the following types of flaws (inaccordance with ASME Code Section XI Appendix VIII)
- Mock-up (or test block) definition and manufacturing



- Development of qualification methodology
- Establishment of the Inspection Qualification Centre.

The mock-ups (test blocks) foreseen are as follows:

- (a) One mock-up (test block) containing WWER nozzle with safe-end including dissimilar weld
- (b) One WWER 440 type RPV mock-up (test block) with circumferential weld and clad/base interface
- (c) One WWER 1000 type KPV mock-up (test block) with circumferential weld and clad/base interface
- (d) One mock-up (test block) of WWER 440 type NPP primary circuit austenitic piping of internal diameter (I.D.) of 500 mm
- (e) One mock-up (test block) of WWER 1000 type NPP primary circuit ferritic piping (with austenitic cladding) of 850 mm I.D.

Experience and know-how of utilities, manufacturers and vendors is planned to be considered in the application of technical justification approach in accordance with ENIQ methodology and used in the Czech approach to inspection qualification. This approach will be applied in conjunction with performance demonstration features according to ASME Code Section XI Appendix VIII.

### **CHARACTERIZATION OF METAL SURFACE BY MEANS OF TWO-DIMENSIONAL FRACTAL ANALYSIS**

S. Horiata, M. Satoh & H. Kitagawa, Toyohashi University of Technology; & T. Tamiya, Kawatetsu Techno-Research Corporation, Japan

Essential property of Fractal is "self-similarity", then theoretical Fractal objects are infinitesimally subdivisible. In other words, those are composed of infinite iteration of an elemental pattern. The structure of Fractal object is described by the "Fractal Dimension" of noninteger. For example, in the three-dimensional Euclidean universe, the dimension of a Fractal object is a noninteger between 2 and 3. In the same dimensional Euclidean universe, the higher Fractal Dimension denotes the more complicated Fractal object.

By analyzing the self-similarity of a material property statistically, we can get several important informations. Fractal method has been applied to inspect the surface flaw of steel sheets. Scanning data detected by a conventional Automatic Surface Inspector was evaluated by one-dimensional Fractal analysis. The results of the analysis was presented [1]. In this study, two-dimensional Fractal analysis is applied to evaluate the surface characteristics of the material. The results are summarized as follows:

- (1) On the calculation of Fractal Dimension, several methods are compared. Simulated data are analyzed by them. From the comparison, the characteristic of each

method is made clear. The cubic box-counting method is proved to be able to detect the subtle irregularity of surface characteristics which can not be found by other usual methods. Then, this method is selected to evaluate Fractal Dimension.

(2) For the measured data, it becomes clear that a certain kind of pre-conditioning must be applied before Fractal Dimension calculation. That is, the data of complicated surface is smoothed by the pre-conditioning in order to lower the dimension. then, Fractal Dimension and Linearity are calculated by the cubic box-counting method. It is concluded from this study that the very subtle irregularity of the metal surface can be clearly detectable by the deviation of both Fractal Dimension and Linearity.

[1] Sixth International Symposium on Nondestructive Characterization of Materials

### **TIP LOCATION OF EXPOSED AND FILLED CRACKS USING MICROWAVES**

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Detection of exposed and filled fatigue/surface cracks on metal surfaces is an important practical issue. Several microwave nondestructive testing approaches have been developed recently for detection and sizing of surface cracks. These techniques include using the dominant or higher order modes generated inside the waveguide (as a result of the presence of the crack). These approaches have shown the potential of detecting filled and cracks covered with dielectric coatings. One important issue associated with these investigations is locating the tip of a crack. This is particularly important from the repair point of view. Rectangular waveguide probes operating in the X- and K-band frequency ranges are used to detect such cracks. The tips are detected once the frequency and the type of the measurement (e.g. dominant mode, higher order mode, phase and magnitude detection) have been optimized. The results of extensive measurements will be discussed in this paper including the accuracy of which the tip may be located. A brief overview of using a rectangular waveguide probe for such measurements will also be presented.

### **COMPARISON OF PARALLEL COMPUTATIONS WITH EXPERIMENTAL VISUALIZATION OF ULTRASONIC WAVES**

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USA; & P. Delsanto, Politecnico di Torino, Italy

Real ultrasonic signals are further complicated by the presence of defects and boundaries in the materials and/or structures. Therefore, understanding the interaction of ultrasonic waves with the defects and boundaries is of great importance for accurate interpretation and presentation of received and modelled ultrasonic signals. An experimental apparatus is described that permits full-field visualization of the stresses inside a material containing defects and boundaries through which ultrasound is propagating.

This relatively inexpensive and easy to use photoelastic experimental set-up has been developed for the visualization of ultrasound in transparent specimens. The experimental set-up employs a light emitting diode as the optical source and a video camera as the receiver. The output of the video camera may be sent to a video cassette recorder or a frame grabbing board for capture of images to a PC for manipulation. The experiment was developed as a means of comparing the results obtained from a numerical model based on parallel computations that we have recently developed to physical experiments. The actual parameters used for the physical experiment are input to the model. Since the parallel computation model can output full-field data, many aspects and details of the computation are readily compared to the experimental results.

### **AUTOMATED SHEAROGRAPHY FOR MEASUREMENT OF RESIDUAL STRESSES**

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One common method for measuring residual stresses in an object is the employment of electrical resistance strain gages together with the hole drilling technique. This method not only requires drilling a hole in the material, but also requires mounting strain gages. These two requirements, especially the use of strain gages, limit industrial applications of this technique for mass inspection in a production environment. Recently, optical techniques such as holography, and shearography are employed to replace the strain gage rosette in the blind-hole drilling method. Although both techniques enjoy the advantages of being full-field, non-contact and not requiring strain gage/transducer, shearography appears to be more practical than holography because it does not require a reference beam. This considerably simplifies the optical setup and most significantly, it eliminates the need of special vibration isolation. However, one major difficulty of using shearography is the fringe interpretation. This difficulty stems from the ambiguities in determining fringe orders and their signs. The reason is that the fringe phase is inherently wrapped into 2 range cycles during formation of the fringe pattern. This paper presents a phase shift technique for automated determination of fringe phase in residual stress measurement.

In this application, an image shearing camera with a large shearing device is employed which brings two images from two separated regions to meet and interfere. Since the deformation due to hole-drilling stress relief is very localized, one of the regions is not deformed and this serves as a reference beam. Thus, this arrangement measures the surface displacements around the neighborhood of the hole. In the testing procedure, the object is illuminated with laser light and it is imaged by the video image-shearing camera. The image of the test object is first digitized and stored in memory of a frame grabber. A hole is then drilled to relieve stresses. Subtracting the deformed image from the stored image produces a fringe pattern depicting the localized displacement around the hole area. Instead of interpreting fringes, the phase shift shearography is used to automatically determine the fringe phase distribution around the hole and the residual stresses are thus determined.

The technique presented provides a fast and automated means of determining residual stresses. It can be used in a field/production environment. Besides residual stress measurement, the method is being extended to the measurement of stresses in large structures such as bridges where live, non-removable loads exist.

## **ULTRASONIC CHARACTERIZATION OF REPAIR PASTES IN CONTEXT OF THEIR BONDS WITH METALS**

M. Josko, Poznan University of Technology, Poland

Since certain time more extensively in technology are used so-called regenerative paste B, for example Belzona molecular metals, Unirep metals, Chester metals etc. These materials are applied for fast emergency repairs by maintenance services in industrial conditions, and for repairing worn or damaged elements of working machines and equipment. The pastes are man-made materials, composed of plastics and metallic fillers. They are combined directly before application, by mixing of two compounds (base and solidifier), in required proportion to a uniform mixture.

The physical, mechanical, and functional properties of the pastes depend on the proportion of the components, roughness of the substrate, temperature, moisture, and other external conditions. Some of the properties are changeable in the time of use. Not all of them are known yet. The majority of them can be evaluated by nondestructive methods, using ultrasonic waves. Selected of them were determined, and the results of measures are discussed in the proposed paper.

In industrial practice such a functional feature as adherence of the pastes to the substrate is an important factor. Usually the adherence is insufficient, and the need of its quality control is essential. Ultrasonic methods enable to evaluate adhesive joints of the regenerative pastes, and the grade of their degradation. In the paper the results of adhesive joints monitoring by ultrasonic methods are presented.

Some of the selected properties of the regenerative materials were considered. First of all the acoustic features, which are necessary to nondestructive assessment bonds type paste/metal were characterized. Directly by the measurement with the aid of the digital ultrasonic gauges, or indirectly, using transformed algebraic expressions, such parameters were determined as sound velocities, attenuation coefficient, acoustic impedance, reflection coefficient, Poisson's ratio, Young's and the bulk moduli. An influence of the testing frequencies was shown in some cases. Using ultrasonic methods, some methodical assumptions were made, and are shown in the paper as well.

## FRACTALS IN NONDESTRUCTIVE EVALUATION

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The lecture objective is to bring attention to new mathematical and geometrical images, i.e. fractals, and their application to nondestructive evaluation. Fractal geometry allows to characterize quantitatively materials and systems with disordered (chaotic) structure, e.g. composites, polymers, colloidal aggregates, rough surfaces and porous materials. Models of such materials and systems, which are based on usual geometrical ideas, turn out to be unsatisfactory. The problem may be solved within the framework of fractal geometry and fractal theory.

Basic ideas of fractals, fractal sets, fractal clusters (fractons) and their quantitative characteristics of fractal dimensions are considered. Traditional examples of fractals, i.e. the Koch curve, the Weierstrass-Mandelbrot curve, the Cantor set and the Serpinsky carpet are given.

Special features of waves scattering by fractals and connection of fractal dimension with angular pattern of scattering are discussed. Data on angular spectra of X-rays and optical waves scattering by internal structure of some polymers and silicon materials are given. It follows out of the data that these materials have fractal structure. Alteration of fractal dimension depending on production conditions of materials formation is discussed. Opportunities to apply fractal dimension to nondestructive evaluation of new materials production are considered.

Chaotic oscillations (chaos) in nonlinear systems have been discovered comparatively recently. One of major features of chaotic oscillations is that they arise in regular nonlinear dynamic systems which are subjected to regular periodic external forces. Chaotic oscillations have an adequate description within the framework of the fractal theory. Strange attractions are fractals. Hydrodynamic and acoustic (cavitation noise) turbulence are the examples of chaotic oscillations.

Fractal measure may be represented by sets with a distribution of fractal dimensions, i.e. multifractals. Data on multifractals and singularities spectra are given. It is noted that they allow to describe illustratively intermittent fields of turbulence. Measurement of near-wall pressure fluctuations in liquid turbulent flows in ducts is described. Experimental data on multifractal characteristics and singularities spectra of wall pressure fluctuations are given.

Singularities spectrum, as differed from power spectrum and correlation function of a process, carries information on the process local structure. This gives more wide opportunities for recognition of signals of different origin. Multifractal analysis gives also reasonable information on different order moments of two-point probability distribution of a process.

Results of experiments are given and applications of fractal analysis for signal processing in nondestructive evaluation (including acoustic emission signals) are discussed. Opportunities for the development of new techniques for defects detection and prediction of material destruction are considered.

### **STRUCTURAL CHARACTERISTICS OF POWDER DEVELOPERS: METHODS OF MEASUREMENTS**

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Hydrodynamic theory of defect's development for liquid penetrant testing has been proposed some year ago. This theory possesses to determine a sensibility of the method when the next structural characteristics of powder developer are known: porosity  $\equiv$ , permeability  $k$  and effective radius of pores  $R_{\text{eff}}$ . Traditional methods to determine these values are not applicable in the case of very thin powder layers of the developer.

In the methods, which have been proposed in this paper, a glass capillary (open or one-side-closed) is used as the basic mean for the measurements.

For the determination of the porosity  $\equiv$  it is necessary to put the capillary with the liquid inside of it's channel in a contact with porous layer. Then we measure two volumes: the volume of impregnated part of developer's layer and residual one inside of the capillary.

To determine effective radius of pores  $R_{\text{eff}}$  for developer's layer we use a glass capillary which is filled with a liquid till prescribed depth and hermetized from one side. If the open side of this capillary is in the contact with porous layer, then liquid is flowing out of the channel inside of the pores, until the equilibrium position of the meniscus when minimum residual depth of filling is reached. Using simple formula for experimental value for initial and residual depth, it's possible to calculate the effective radius of pores or powder layer  $R_{\text{eff}}$ .

Preliminary determination of  $\equiv$  and  $R_{\text{eff}}$  for the determination of permeability  $k$  is required. The formula for the calculation of  $k$ , which contains the properties of liquid (viscosity and surface tension), porosity  $\equiv$  and effective radius of pores  $R_{\text{eff}}$  is derived.

The results of this paper give an opportunity to describe theoretically both hydrodynamics of defect's revealing and the sensibility of liquid penetrant testing for given penetrant-developer set.

## **ON THE MATTER OF PHYSICAL NATURE OF THE SO CALLED LONGITUDINAL SUBSURFACE WAVES**

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This work is devoted to the theory and practical applications of so called longitudinal subsurface waves for ultrasonics and acoustic methods of nondestructive techniques which are in active discussion during the last ten years. Considerable differences in approaches to understanding of the physical nature of these waves and terminology of their definition, as well as to the measuring techniques are analyzed. It is mentioned that presently existing recommendations for their practical applications for nondestructive evaluation of materials are not sufficiently developed. A new insight into the physical nature of these waves is suggested on the base of a new investigation of Rayleigh equation. Their main characteristics connected with numerical solution of the Rayleigh equation are described. The formulae for calculation of elastic constants of materials using values of velocity of longitudinal subsurface waves and other types of surface and bulk waves are given. Some results of experiments confirming this new approach are presented in the work. A presumption of existence of a new type of waves which is based on a solution of the fundamental Rayleigh problem is proposed. It is suggested that further investigations in this direction contain possibilities for development of new effective techniques for nondestructive materials characterization in future.

### **MAIN PHYSICAL CHARACTERISTICS OF LIQUID PHASE DEVELOPERS**

P. Prokhorenko, A. Kornev & I. Stoicheva, Belarussian Academy of Sciences, Belarussia

Liquid developers: suspensions, colloidal solutions, paints, lacquers - have been widely used in the penetrant testing due to its technological advantages. We have shown [1,2] that interaction of the liquid phase of the developer with the penetrant in the cavity of the defect considerably influences on the process of liquid extraction from the defect into the layer of developer at the surface, which is directly linked with sensitivity of the penetrant testing. So, it should be taken into account, that the liquid phase of the developer is not only the passive bearer of the developing component, but also actively influences on the process of developing.

Experimental data show that when one liquid partially fills the dead end capillary channel, and the second one contacts the former at the channel entrance, the following results of liquids interaction are possible: full extraction of the liquid from the capillary channel, partial extraction, increase of filling depth. It has been established that the interaction mechanism is determined by change of the end angle of washing, the coefficient of the surface tension on the inside meniscus of the liquid and change of pressure of saturated vapors, in the gas closed in the top, as the result of mixing and diffusing both liquid phases. As the result the criterion was elaborated which took into account change of

these physical and chemical parameters and geometry of the capillary channel:  $\Delta P_v - \Delta P_c > 0$  where  $\Delta P_v$  - difference of pressures of saturated vapor of liquids,  $\Delta P_c$  - difference of capillary pressures, created by liquids in the capillary channel. Pressure of the gas and vapor mixture, closed in the dead end of the capillary is the sum of partial pressures of air and saturated vapor of the liquid phase. In the process of interaction of these liquids this pressure can be changed by the value, which is determined by the difference of saturated vapors pressures of pure liquids. Factor  $\Delta P_v$  reflects this phenomenon. Geometrical parameters of the capillary channel are inobviously taken into account in factor  $\Delta P_c$ .

In case of execution of inequality the result of liquids interaction will be fully or partially extraction of liquid from the capillary channel, depending on the difference value. If the inequation is not true, the degree of capillary channel filling is increased. So, at the beginning, when the liquid developers are used, the process of developing proceeds on the base of interaction of the liquid phases of the developer and the penetrant.

Account of interaction of liquid phases and use of criterion in practice make it possible to purposefully choose substances when preparing flaw detection materials and sets for penetrant testing. The given effect is important not only in the field of the penetrant testing but in the broad spectrum of processes: cleaning, impregnation, lubrication, extraction, corrosion prevention, membrane technology, etc., where liquid media are in contact into bodies having capillary porosity.

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## **THE ESTIMATION OF ELASTIC MODULUS OF METALLIC MATERIALS BY DYNAMIC INDENTATION METHOD**

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The known ultrasonic and static indentation methods of the elastic modulus measurement are very complex in the realization. We propose the method of material testing based on the registration of the dynamic indentation process. Taking into account that used impact energy is not more 2-3 mJ damage of tested surface is insignificant (residual depth of the indentation is no more than 15-20 micrometers) this method can be classify as nondestructive.



During impact interaction of the spherical indenter with the tested material elastic-plastic deformation (loading stage) and truly elastic one (unloading stage) take place. The recovery of elastic strain in the unloading stage gives the based information for the calculation of the elastic modulus. On the base of Hertz's theory with reference to the unloading stage of the impact we obtain the expression for  $E_1$  as follows:

$$E_1 = \frac{1 - \mu_1^2}{2 \sqrt{D \cdot y_{\max}} / (|P|/y) - 1 - \mu_2^2} / E_2$$

where  $\mu_1$ ,  $\mu_2$  and  $E_1$ ,  $E_2$  are the Poisson coefficients and the elastic modulus of tested material and indenter respectively,  $D$  is the diameter of the indenter spheric tip,  $y_{\max}$  is maximum displacement of the indenter,  $|P|/y$  is the slope of the unloading curve in the point  $y_{\max}$ .

The test apparatus consists of the indenter attached to the rotation lever edge, velocity sensor, gauge amplifier, digital memory and computer. The original information about the motion of the indenter during test impact is a file of motion velocity of the indenter. Data of dynamic load, depth of the indentation and the calculation  $E$  are obtained by numerical methods according to the program by the computer.

The apparatus measures elastic modulus in the local area of volume  $0.5 \text{ mm}^3$ . The testing results of a different materials are in a good agreement with a data of ultrasonic methods.

## ULTRASONIC CHARACTERIZATION OF BURRS IN AI-PRESSURE CASTINGS

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Hollow castings are semifinished products of high functional versatility. They are usually superior to components made by other jointing methods in terms of strength, toughness, weight reduction and economic production.

The burrs, caused by the jointing process of the different branches of the molten material passing through the matrix, can be areas of poor mechanical properties. Reduction in strength and toughness and an inferior fatigue behavior are the major drawbacks. Up to now the quality of the burr is checked by destructive sampling; the nondestructive characterization of burrs is a considerable step towards the quality assurance concept. The contribution presents results of an experimental investigation.

Ultrasonic backscattering technique was used to visualize the fine grained structure of the burr area, in between the areas with larger mean grain sizes and higher contents of

impurities. Comparing the results with the optical photomicrographs the significant change along the length of the pressed part is confirmed.

The texture in the burr area and in the adjacent parts have been found to be different and this difference varies along the length. The expansion coefficients of the orientation distribution function were calculated using X-ray pole figures taken at different positions and under different directions. Using the expansion coefficients, the texture influence on the ultrasonic velocities were calculated and compared with the measured results.

Both techniques were found to be suited to determine this position along the length of the casting where the burr reaches the optimal mechanical properties.

### **ULTRASONIC CHARACTERIZATION OF TEXTURE IN ALUMINUM ROLLED PRODUCTS**

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Germany

The texture in rolled Al-sheets has a strong influence on the material behavior during deep drawing process. A wrong texture causes unacceptable earing. The state of the art is the destructive texture analysis using the samples cut from the manufactured sheet.

As a step towards nondestructive quality assurance, an ultrasonic prototype system has been built and tested using samples from a hot rolled strip. The time-of-flight of an ultrasonic SH-wave was measured as a function of the wave propagation direction with respect to the rolling direction. The measurements were performed at the center of the width of the strip, at the edge and in between these two positions. The difference between the two extreme values of the time-of-flight profile, measured at the center position was used as a measure for the earing. There was an excellent agreement between the ultrasonic result and the destructively evaluated earing. Furthermore, inhomogeneity in the texture along the width of the strip was detected.

The expansion coefficients of the orientation distribution function were used to calculate the influence of texture on the SH-wave velocity as well as on the velocities of other wave modes.

The contribution presents the experimental results and discusses the advantages of the techniques used for on-line application.

## **APPLICATION OF MAGNETIC BARKHAUSEN EFFECT FOR EVALUATION OF STRESSES AND STRUCTURE OF FERROMAGNETS**

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In a time the Magnetic Barkhausen Effect (MBE) is well known as an effective and perspective method for diagnosis and nondestructive testing of structure and stresses in surface layers of ferromagnetic materials. At the same time there are some problems and the main is the problem of increasing the selectivity (selective sensitivity) of the measured MBE to the stress and structure parameters under testing.

In conventional report some techniques, technical means, algorithms and softwares, which realized in the created system INTROMAT with extremely high selectivity, are discussed.

They are following:

1. High sensitive sensors, which allow to test the components with complex shapes and surfaces (fillets, intrinsic surfaces of holes, convex and concave surfaces etc.) and with anisotropy of properties.
2. Technique and software for separate evaluation of stresses and structure, based upon the separation of anisotropic and isotropic parts of the signal.
3. Technique and software to select optimal regimes of testing, having the highest sensitivity to the parameters under testing.
4. Technique for getting the calibration curves, which allow to account the influence of residual plastic deformation on the results of testing (magnetic-plastic hysteresis loops).

Their implementation in INTROMAT exhibits reliable nondestructive evaluation of degree of strengthening after heat treatment, cold rolling, chemical and thermochemical surface treatment, laser beam quenching, to measure the stress tensor components for plane stress state, to test phase content in steels (e.g. austenitic content), chemical components content (e.g. carbon content), to detect burns, for steels sorting etc. The system INTROMAT and the techniques are used on the aircraft and shipbuilding plants, in automobiles industry etc.

## COMPARISON OF ABSOLUTE SENSITIVITY LIMITS OF VARIOUS ULTRASONIC AND VIBRATION TRANSDUCERS

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Previous work has compared the relative performance of various wide-band ultrasonic transducers used as receivers [1]. Studies have also been made comparing the relative performance of various optical sensors [2] and evaluating their applicability as acoustic emission (AE) [3] sensors. In this paper, the calculated and measured sensitivities of such transducers are compared with the sensitivity of transducers capable of detecting low amplitude AE events in steel. While optical sensors appear to provide many practical advantages over contact sensors, particularly at very low frequencies, it is found that they cannot meet the sensitivity requirements for wide-band AE studies in metals. Furthermore, it is found that a new transducer, based on a refinement of the NIST secondary-standard conical transducer, has sufficient sensitivity for such applications. In particular, this new, high-fidelity, high-sensitivity sensor is found to exhibit absolute sensitivity which approaches the "thermal rattle" limit in aluminum within 10 dB in the 250 kHz to 1 MHz region. Also, it is shown that the new transducer's noise floor is well below both the sensitivity level needed to monitor AE in metals [4,5] and the noise floors of both optical and airborne-sound transducers. Furthermore, its performance is in good agreement with the computer model used in its design.

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## **BEHAVIOR OF CONCRETE OBSERVED BY ACOUSTIC EMISSION MEASUREMENT**

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Behavior of concrete under different load conditions will be considered on the basis of laboratory test of plain and reinforced specimens. Acoustic Emission was found as the efficient technique for better recognition of concrete characteristics.

Laboratory tests were carried out in several groups:

- test under compression-impact, static and repeated loading cuboidal and cylindrical specimens made of plain concrete, small reinforced concrete specimen with different number and location of reinforcement.
- test of reinforced concrete beams under bending

The main goal of tests better qualitative description of fracture process in concrete in relation to mode of applied loading and positioning of reinforcement. Test program was also design for better understanding of the AE phenomena in concrete.

In conclusions some general remarks on fracture of concrete in the view of hypothesis of microcraks cumulation will be shown.

## **CRACK CLOSURE DURING CYCLIC FATIGUE IN MG-PSZ CERAMIC AS DETECTED BY ACOUSTIC EMISSION**

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Crack closure is a well recognized phenomena during cyclic fatigue of metal materials. The effect of crack closure is to cause fracture surface contact above the minimum load of the loading cyclic during cyclic loading. Crack growth during cyclic loading,  $da/dN$ , is dependent upon the applied stress intensity factor,  $(K=K_{max}-K_{min})$ , according the Paris Law,  $da/dN=A((K)^n)$ . When crack closure occurs ( $K$  is reduced to effective applied stress intensity factor,  $(K_{eff}=K_{max}-K_{cl})$ , where  $K_{cl}$  is the stress intensity factor at which crack closure occurs.

Crack closure detection is traditionally undertaken by observing compliance changes during cyclic loading via a strain gauge attached to the back face of the test sample behind the crack tip. In ceramic materials the strains associated with cyclic loading are very small and compliance changes, and hence crack closure, are almost undetectable by the use of back-face strain gauges.

In this work a piezo electric acoustic emission detector was placed on the top of a compact tension sample above the crack tip. The material used was a magnesia stabilized partially stabilized zirconia (Mg-PSZ). The acoustic emissions involved with fracture surface contact were used to detect the occurrence of crack closure by simultaneously recording the applied load at the same time as the acoustic emission. Tests were undertaken inside a scanning electron microscope to allow direct correlation between acoustic emissions and crack profile observations.

Two types of crack closure were detected: the first occurs near the crack tip at relatively high stress intensity factors and is proposed to be due to dilation of the material near the crack tip as a result of stress induced phase transformation. The second occurs at lower stress intensity factors, and hence further behind the crack tip, and is proposed to be due to fracture surface roughness and/or asperities between the crack faces. The former type of crack closure near the crack tip is considered to be the most significant as the cyclic fatigue degradation mechanism in Mg-PSZ is believed to be an intrinsic involving processes at the crack tip.

Crack closure at the crack tip would therefore be a significant factor affecting cyclic fatigue in phase transforming ceramic materials.

## **ANALYSIS OF THE ULTRASOUND SIGNAL ACCORDING TO THE CREEP-RESISTING MATERIALS USED IN ENERGETICS**

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This report concerned with frequency analysis of the ultrasound signal on the samples from the creep - resisting steels used in energetics. This method can be used for the need of continuous diagnostics of the selected energetic plants.

The results show that with suitable selection of the informative parameters of the ultrasound signal, it is possible to follow up the steel features mentioned above.

## **PLASTIC AND ANPLASTIC BEHAVIOR OF ZIRCONIUM POLYCRYSTALS**

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The investigation of the internal friction in crystalline solids is used to give information about dislocations, point defects and their interactions. In order to obtain information on low temperature changes of acoustic properties of alpha zirconium we have measured the temperature dependence of Young's modulus and internal friction in polycrystalline zirconium samples in the temperature range from 6 to 320K. Alpha zirconium polycrystals were investigated by resonant frequency method at 87 kHz.

Young's modulus  $E$  and decrement measurements were performed on undeformed and deformed samples. The present investigation demonstrates that the temperature dependence of the dynamic Young's modulus  $E$  cannot be described by a simple linear function over the whole temperature region studied. After plastic deformation the modulus defect  $\Delta E/E_0$  is also observed. From the experimental data on relaxation peak in damping at 255 K the activation enthalpy has been evaluated. The results are discussed in terms of current models.

### **STRESS RELAXATION OF SHORT FIBRE REINFORCED Mg METAL MATRIX COMPOSITES AFTER PLASTIC DEFORMATION DUE TO THERMAL CYCLING**

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Cooling of metal matrix composites (MMC) from solidification temperature to room temperature causes inherent stresses due to the difference between the thermal expansion coefficients of the MMC components. Thermal stresses above proof stress lead to plastic deformation of the metal matrix in the absence of external forces. Increased temperature decreases the yield point and accelerates creep and stress relaxation of the matrix. For this reason the plastic deformation depends on the time-temperature-profile that is applied to the MMC. The reversible stress relaxation is very sensitive to microstructural changes that take place at the matrix-fibre interface of the thermally cycles MMC's.

The conventional cast alloy AZ91 (nominal composition 9wt.%Al, 1wt.%Zn, 0.2wt.% Mn) and the creep resistant alloy QE22 (nominal composition 2.5wt.% Ag, 2wt.%Nd, 0.6wt.% Zr) both reinforced with 22vol.%Al<sub>2</sub>O<sub>3</sub> short fibres have been investigated. The short fibres are planar isotropically distributed. Specimens for reversible stress relaxation measurements were taken parallel to the reinforcement plane, solution heat treated and artificially aged. The influence of the thermal cycling on the relaxation behavior of the specimens is varied by the preceding heat treatment, the maximum temperature, the number of cycles and the ageing time at room temperature.

The irreversible relaxation of the internal stresses due thermal cycling has been investigated by dilatometric and internal friction measurements and microscopy. The experiments characterize the internal stresses of MMC's due to the effects of their relaxation.

## **DETECTION OF MICROSTRUCTURAL CHANGES AND INTERNAL STRESSES OF MMC'S BY STRESS RELAXATION MEASUREMENTS**

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Nondestructive testing and characterization of most materials can be performed by the measurement of their internal friction. It is well known that various properties of internal friction like the damping capacity or reversible relaxation strength are most sensitive on microstructure.

In nearly pure metals a strong dependence of the internal friction on the concentration of solid solutes has been observed which in many cases can be attributed to an interaction between them and dislocation segments. This also holds for metal matrix composites (MMCs) with a low alloyed phase in the metal matrix.

The reversible time dependent stress relaxation strengths of three various MMCs depending on preceding heat treatments were measured at room temperature. Two of these MMCs were saffil-fibre reinforced magnesium alloys cp-Mg/20%-Al<sub>2</sub>O<sub>3</sub> and AZ91/20%-Al<sub>2</sub>O<sub>3</sub> produced by melt infiltration. One MMC (Dispal) was powder metallurgically produced aluminum with silicon and carbide dispersions.

In all three materials an aging effect at room temperature was found. This means, that the relaxation strength is highest immediately after heat treatment and decreases to a constant value in one to two days.

A linear increase of the relaxation strength measured immediately after isochronal heat treatment with its temperature is found in cp-Mg/20%-Al<sub>2</sub>O<sub>3</sub> and in Dispal, the two MMCs with nearly pure metal matrix. This increase ages out nearly completely in one day. In AZ91/20%-Al<sub>2</sub>O<sub>3</sub> the relaxation strength is maximum, when the precipitation in the matrix is complete and the concentration of solutes in the matrix is minimum.

All these experimental observations can be used for NDT. They can be explained by solid solutes preventing dislocations from bowing out. Free dislocation segments are produced by thermal stresses due to the different thermal expansion of the metal matrix and the reinforcements.



## **LOW-TEMPERATURE INTERNAL FRICTION IN NIOBIUM OF DIFFERENT PURITY DUE TO MOTION OF GEOMETRICAL KINKS IN DISLOCATIONS**

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Low temperature dislocation relaxation phenomena are studied in niobium in a wide range of the impurity content. The acoustical measurements have been carried out at the frequencies 50..350 kHz by the two-component oscillator technique in the temperature range 2..12 K. Large sound absorption peaks and modulus defects are detected after rapid cooling of samples in both the normal (n) and superconducting (s) states. Surprisingly, in the s-state the anomalies are observed at higher temperatures than in the n-state. Influence of impurity content, orientation, cooling rate and plastic deformation on the acoustic anomalies has been investigated. Purification of the samples and/or preliminary plastic deformation at room temperature suppress the sound absorption peak and corresponding modulus defect. It is supposed that the great number of geometrical kinks in screw dislocations are generated during rapid cooling of samples. In this case thermally activated overcoming the 2nd order Peierls potential barriers by the geometrical kinks is responsible for the anomalies observed. Activation parameters of the geometrical kink motion are established. The results obtained may be used when designing cryogenic devices with high mechanical quality factor (resonant-bar gravity-wave antennas etc.).

## **ACOUSTOPLASTIC EFFECTS IN CRYSTALS**

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A brief review of acoustoplastic effects (APE) is presented. In general, there are two types of these effects: during creep superimposed vibrations may cause an increase of creep rate (Archbutt effect) and during active plastic deformation they may decrease the static flow stress (Blaha-Langenecker effect). The most developed theories of APE are phenomenological. They treat APE only as a result of superposition of static and alternative stresses. In this case APE is predicted only if there is a non-linear relationship between the strain rate (or dislocation velocity) and the stress. The physical nature of APE still remains obscure, although several mechanisms have been proposed:

- (1) a local heating under influence of ultrasonics,
- (2) changes in the defect structure,
- (3) a re-distribution of internal stress fields.

It is difficult to detect residual structural changes because during APE, especially at low amplitudes of vibrations, these changes are reversible. That is why internal friction (IF) techniques have been used in recent years [1-4]. In [2-4] the attenuation of ultrasonic waves causing APE was recorded. The results show that APE can not be explained by the

superposition mechanism only. If experiments clearly indicate that APE can not be attributed to a heating of the sample under ultrasonics. On the other hand, there is evidence for reversible changes of dislocation structure. The internal stresses also may be affected by superimposed vibrations. These mechanisms are discussed within the framework of available theories and experimental data.

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## **EVALUATION OF DILATANCY IN ROCK FOR A FORECASTING OF BURST-PRONE ZONES IN MINES**

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Rock samples were tested with the use of hydraulic test machine to investigate dependence of released elastic energy on a size of occurring defect. Acoustic emission signals generated at the defects' formation during loading and deformation of samples were registered by the wide band piezoreceivers. Strain measurements were carried out as well. An invariant co-relation between the defect size and released energy was obtained. It was also shown that dilatancy is changing in time and this feature could be used for a forecasting of rock failure events of different scale levels. The obtained dependencies make it possible to evaluate a dilatancy in locations of rock massif which are unavailable for direct observations and doing so to predict rock bursts. A relatively simple microseismic network that can be used for the rock mass monitoring is also discussed.

## **ENERGY-FREQUENCY DISTRIBUTION OF ACOUSTIC EMISSION FROM LOADED ROCK SAMPLES**

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An acoustic emission (AE) activity was recorded during the loading of granite rock samples of different grain size by uniaxial compressing load. Wide band recording system with high dynamic recording range enables to analyze individual acoustic emission events in a wide frequency (10 KHz to 1 MHz) and energy range (more then 120 dB). The inner structure of individual samples was determined with the help of precise petrological sample analysis. The structure was described by the grain distribution of individual minerals. Acoustic emission monitoring enables to study energy-frequency distribution of AE events,

that could be fitted by negative exponential distribution -  $N(E) = aE^{-b}$ , where  $N(E)$  is the number of events with energy  $E$  and  $a$ ,  $b$  are the distribution parameters;  $a$  - additive constant and  $b$  - the slope of decreasing line in bilogarithmical co-ordinates. The experimental data analysis proved that above mentioned dependence is not valid for the whole energy range. Clearly pronounced deviation was observed mainly for outer parts of the distribution-for weak and strong AE events. It was proven that energy-frequency distribution of AE events radiation from loaded rock samples strongly reflects the inner structure of the samples, described by the distribution of structure grain size. Minimum dimension of grains influence the lower part of energy-frequency distribution; upper part of the distribution (higher frequencies) is limited by the size of the sample. Typical energy value of AE event (most frequently radiated amount of AE energy) reflects most frequently observed grain dimensions.

### **KINETIC APPROACH TO THE NONDESTRUCTIVE MONITORING OF ROCK FAILURE**

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Method of physical kinetics is proposed as a basis for the monitoring of the process of rock failure in laboratory and field experiments. Kinetic curves showing dependence of the cumulative number of acoustic emission signals vs time were obtained with the use of test machine for different kinds of rock samples to demonstrate a possibility of the proposed approach to separate the different stages of the rock failure process. The transfer from 3D to 2D solution in case of pre-failure crack formation was used for forecasting of rock failure event including rockburst. The rockburst data which have been obtained for several mining areas were reprocessed to demonstrate the possibilities of the kinetic method. As was shown the proposed method allows one to create a monitoring system capable to separate natural and manmade seismic events that is of a big importance for a safe mining.

### **ACOUSTIC EMISSION IN AMORPHOUS METALS**

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Russian Academy of Science, Russia

A low macroscopic plastic deformation of amorphous metals (AM) at the room temperature is one of the most prominent features and one of the most important characteristics of this kind of materials. However, it is known that the plastic deformation may be extremely large in the small local volumes of AMs. The problem of characterization of plasticity in AM is far from the final solution by now. Therefore, there still a great deal to be learned about the mechanisms of plastic deformation of AM. The application of a modern acoustic emission (AE) technique to the problem mentioned is discussed in the present work. The transient AE signals of a relatively high energy have been detected

during tensile testing of Co and Fe based metallic glasses. It is shown that a microscopic plastic deformation of amorphous metals is activated long before the failure. This process is manifested as the quite inhomogeneous evolution of defects of different scales. At least three scale levels of inhomogeneous plastic deformation differed by the degree of temporal and spatial correlation can be discriminated by means of statistical and correlation AE analysis including a detailed examination of shape and a frequency spectra of individual pulses. 1) In-situ experiments performed in scanning electron microscope together with AE measurements have clearly shown that the appearance of a shear band on a free surface is accompanied by the AE signal of a specific shape. 2) Using a statistical approach it is shown that after the appearance of a few number of independent slip lines the shear bands interact with each other. It leads to the correlated AE pulse flow as well as the formation of the ordered systems of shear bands on the surface of the specimen. Such a co-operative behavior of the shear bands strongly depends on a chemical composition and mechanical properties of material. 3) Third level of microscopic plastic deformation in amorphous metals is reflected by low amplitude AE having a specific spectrum differed from that in the case of shear band formation. It allows to conclude about the activation of certain mechanisms of plastic deformation despite the fact that they were not detected by optical or scanning microscopy. It is supposed that those processes are connected with slip formation. It is shown that they frequently precede the appearance of shear band on the surface.

### **ACOUSTIC EMISSION (AE) AS A TOOL FOR MONITORING THE ELECTRICAL, THERMAL AND ELECTROMAGNETIC EFFECTS DURING THE BRITTLE CRACKING OF CERAMIC MATERIALS**

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The AE signals caused by the micro-cracks appears in the early stage of cracking process, when the other methods are not sensitive enough. For this reason the AE method has a unique value for non-destructive testing. Simultaneously with AE activity the mechanical load cause in ceramic materials the changes of the electrical conductivity and generate the electromagnetic emission. The correlation of these effects was investigated. The diagram of conductivity changes versus applied force has the linear segments in the initial part related with AE count rate increase. This give the possibility to forecast the critical strength and the "life time" of the material from the moment of appearance of the first group of AE signals. The acoustoelectric coefficient  $Q$  is define as the ratio of electrical conductivity change of AE count rate for two different mechanical loads. The influence of the thermal shocks during long-time operation of the high voltage insulators was also investigate. A dependence between the temperature changes and the AE count rate was stated.

## ACOUSTIC EMISSION ANALYSIS OF GRAIN BOUNDARY EFFECT ON PLASTIC DEFORMATION IN BICRYSTALS

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The role of grain boundary (GB) in acoustic emission (AE) is examined during tensile deformation of the [110] oriented Cu and Cu-Al bicrystals having  $\{9(221)\}$  longitudinal tilt boundaries and of the single crystals of the same orientation. Features of AE spectra in stages I, II and III of plastic deformation are shown. Results are discussed from the viewpoint of the co-operative behavior of defects. They can be summarized as follows.

1. The refined effect of GB on AE is established. The following features of AE related to the presence of GB have been found via systematic AE analysis including the analysis of energy spectra.
  - the AE-level in Stage II of deformation of bicrystals is significantly larger than that for component single crystals.
  - A remarkable increase of the low-frequency narrow-band spectral component take place at the end of Stage II and during Stage III of bicrystal unlike the monotonous shift of spectra toward the high frequency domain in the single crystal.
  - The appearance of a few number of wide-band noise-like spectra in bicrystals has been detected in bicrystals in contrast with single crystals showing much more uniform AE. These features are interpreted from the peculiarities of dislocation structure and its behavior near the GB.
  - The GB plays a role of dislocation source which gives a contribution to a cumulative AE due to the formation of the secondary slip close to GB. These sources have no substantial spectral features in comparison with AE sources in single crystals.
  - The raise of low-frequency component in AE spectra of bicrystals is due to the coarse slip which forms as a result of pileup movement after overcoming the GB.
  - Noise-like spectra in bicrystals can be associated with the blunting of pileup passed through the GB.
2. A kinetic model of pileup movement after cutting the GB proposed in the present article satisfactory explains the appearance of specific AE pulses at the end of Stage II and during Stage III of deformation as well as a coarse slip formation close to GB.
3. A statistical approach to the characterization of defect ensembles is found to be fruitful for evaluation of microscopic parameters of defect dynamics and kinetics. Estimations of a dislocation mean free pass and a flight time have been done from the AE spectral data.

## **DAMAGE MONITORING DURING MONOTONIC TENSILE LOADING OF QUASI- ISOTROPIC CARBON/EPOXY LAMINATES WITH THE USE OF ACOUSTIC EMISSION TECHNIQUE**

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This paper describes the damage development in quasi-isotropic Carbon/Epoxy laminates, under monotonic tensile loading. The correlation between the applied strain and the onset of different damage types has been established using different non destructive test techniques.

The tensile tests were carried out on a quasi-isotropic  $[0, \pm 45, 90]_s$  laminate, Ciba Geigy Fibredux 6376C-HTA-5-35, and the damage monitoring was performed using the edge replication and the acoustic emission techniques. Main emphasis was put on the discriminating power of the acoustic emission method to monitor the individual damage types during the tensile test. Two different strain rates have been tried out. The replication technique revealed the following damage development at the edges of the specimens:

- transverse cracking of the  $90^\circ$  plies until the Characteristic Damage state (CDS) is reached.
- delamination in-between the  $90^\circ$  ply and at the  $90^\circ/-45^\circ$  ply interfaces.
- increase of the opening of the edge delamination and propagation along the length of the specimen.

The final failure of the specimen is expected after an accumulation of fibre failure in the  $0^\circ$  plies, due to high stress concentrations developed along their length.

Based on the peak amplitude distribution of the acoustic emission signals, it was decided that unstable crack propagation in the  $90^\circ$  plies, resulting in full width matrix cracks, produces high amplitude signals. Lower amplitude events occur when the crack propagates in a stable way. Delamination covers the medium range of the peak amplitude. The fibre failure amplitude range has to be investigated further on, but based on the present results, it seems that fibre failures in this composite material do not produce high amplitude events.

## **APPLICATION OF INTERNAL FRICTION AND ACOUSTIC EMISSION METHODS FOR OF MACHINE MANUFACTURING MATERIALS PROPERTIES**

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Assurance of machine manufacturing articles reliability depends on opportune and reliable evaluation of materials essentially, mechanical systems state and predicting of changes during time. Internal friction (IF) value relates to microstructural heterogeneity functionally, density and distribution of defect in material microstructure. It is used for articles monitoring at various technological processes stages of their manufacturing. Method of acoustic emission (AE) gives the fullest information about the changing of materials structural condition under exterior loadings. Method of AE is effectively used for timely evaluation of heavily loaded parts and friction surfaces state.

The results of theoretical and experimental investigations of friction pairs, made with the IF and AE methods, are generalized.

The application of information about energetic fluxes distribution explains the causes of structural conditions changing, contacting surfaces strengthening, intensifying-adhesion and diffusive processes in the thing surface layers of contacting materials. It allows to define the thickness off defective surface layer.

The possibility of layered materials with demanded quality parameters creation is substantiated. The methods of materials matching for friction units are described.

## **RECENT DEVELOPMENTS IN REAL-TIME ACOUSTO-ULTRASONIC (AU) NDE TECHNIQUE TO DETECT & MONITOR VARIOUS DAMAGE MODES**

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The acousto-ultrasonic NDE technique is a hybrid of the acoustic emission (AE) and ultrasonic methods. The AU technique excites a repeated series of ultrasonic pulses in the specimen by a broad band transducer through a couplant medium. A receiver transducer detects the excited stress wave pulse after it has interacted with the material micro-structure and the damage present between the sender and the receiver transducers. Analysis of the received signal in the frequency domain produces a stress wave factor (SWF) which can be correlated to residual strength/stiffness of the specimen through additional experimental measurements.

A feasibility study of real-time AU NDE technique to detect and monitor damage in composites under static/fatigue loadings was accomplished. Real-time AU was found to

detect and monitor the growth of matrix cracking, fiber breaks, longitudinal-splits and micro-buckling. Continuous video recording of the damaged surface corroborated the conclusions of AU data. This paper summarizes the work done till date by the authors on development of AU to detect/monitor various damage modes occurring in composites and thereby help in mechanical characterization. Unlike this real-time AU technique, other NDE methods usually require interruption of the mechanical test to evaluate damage state. Continuous monitoring of damage by real-time AU NDE technique has enhanced our understanding of damage mechanisms in composites.

### **JOINT-TIME-FREQUENCY-ANALYSIS OF ACOUSTO-ULTRASONIC WAVEFORM DATA**

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When coupled to statistical or neural pattern recognition classifiers, one inspection technique showing significant promise in both mechanical performance prediction and rapid part throughput of complex-shaped composite material components, is Acousto-Ultrasonics (AU). Large-scale acceptance of AU by the NDE community has not occurred because the operation of a pattern classifier that can correctly sort materials, is not evident to most in the community used to dealing with specific ultrasonic propagation phenomena interacting with material state.

The subject of this paper, and an important aspect of the AU methodology, deals with the analysis of AU data. In general, AU signals are too complex to analyze with the naked eye; without computer-assisted intervention. An AU signal consists of a superimposed collection of transient waves with varying arrival times, durations, and frequency content. The resulting signal is very much a function of the form of insonification (e.g. transducer positions), the scattering geometry/part shape, the degree of anisotropy, the stimulus and, most importantly, the state of the material (or adhesive region) being characterized.

This very complex one dimensional signal is first transformed into a time-frequency format. The AU signal analysis problem can then be somewhat standardized and demystified because of added propagation mode visibility in time and frequency, where the ultrasound/material interaction, that is critical to the underlying discrimination process, becomes more evident. The utility of neural and statistical pattern recognition approaches applied to various joint-time-frequency representations of AU signals are investigated, with an emphasis on: discriminating information location, time-varying parametric spectral estimates, phase-sensitive spectral estimates, as well as time-varying filter-based distance functions for real-time, on-line implementations. These methods are examined using AU signal data previously acquired from a thick composite structure being produced in a real manufacturing environment.



## **COMPUTER SIMULATION OF ACOUSTIC WAVES PROPAGATION IN ELASTICALLY ANISOTROPIC MATERIALS**

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Computer simulation for elastic waves and its applications to acoustic waves in elastically anisotropic materials are presented. The simulation is based on a finite difference method and a mass particle method, using a newly developed procedure for calculation of acoustic particle displacements under fundamental considerations of elastic wave equations. In the present method, elastic anisotropy is represented by stiffness matrices, and various combinations of materials with different elastic properties are allowed.

Its computer program is designed for 3-dimensional calculation, so that the applications include 3-dimensional particle displacements. The method has still a problem in calculations of anisotropic materials with elastic properties of complicated stiffness matrices. However, the calculation succeeds to visualize the complexed wave propagation in hexagonal and cubic crystals, a unidirectional fiber reinforced material, and a multi-layered material, which show elastic anisotropy macroscopically. In the visualization, distributions of acoustic group velocities in the crystals are directly observed, and show a good agreement with theoretical ones.

## **A STUDY OF LAMB WAVE INTERACTION WITH DEFECTS IN THIN POLYMER AND METALLIC MATERIAL USING A DIFFERENTIAL FIBRE-OPTIC BEAM DEFLECTION TECHNIQUE**

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Ultrasonic Lamb waves have been extensively used for the nondestructive evaluation (NDE) of plates, pipes and laminates. Lamb wave modes can be selectively generated using conventional transducers, operated in a pitch-catch or pulse-echo arrangement, requiring either immersion coupling or contact to the test specimen. Thermoelastic line sources from a pulsed laser, producing symmetric ( $S_0$ ) and asymmetric ( $a_0$ ) Lamb waves, have also been studied in aluminum sheet and polymer film of varying thickness. Such transient waveforms have been processed, using the long wavelength approximation, to calculate the elastic properties of the plate under inspection.

More recently artificial notches have been machined into metallic and polymer samples, to simulate defects. Studies in both time and frequency domain have examined their effect in propagating Lamb waves. In defective plates an additional Lamb wave mode has been observed, with similar temporal and amplitude characteristics to those of the frequency - dispersive  $a_0$  mode. It arises from mode conversion of the  $s_0$  mode by the defect.

For non-contact testing applications laser-ultrasonic based systems offer advantages of truly remote and point detection. In our experiments, a flexible fibre-optic sensor was used for the detection of these thermoelastically-generated Lamb waves. The optical detection system consisted of a single mode fibre which delivered a 14mw probe spot on the sample surface. Angular perturbations of this spot due to ultrasonic transients were monitored using a fibre-optic bundle incorporating differential photodetection. The sensitivity of the system was sufficient to enable the detection of Rayleigh waves reflected from artificial defects as well as signals from mode converted Lamb waves.

Results demonstrate, that for thin 125 m thick, metallic specimens, mode-converted Lamb waves can be clearly distinguished from defects, which range in depth from 10 m to 40 m. These measurements can be made on a single shot basis.

#### **OBSERVATION OF INTERNAL DEFECT IN FUNCTIONALLY GRADIENT PSZ-Ni BY ULTRASONIC IMAGING**

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Functionally gradient Material, FGM, is a ceramic-metal composite of which structure is changing gradually to decrease a thermal stress between ceramic and metal bonding interface while used at high temperature. Although an ideal FGM is consisted of continuously changing composition, a laminated type FGM is widely studied because it is produced easily. The laminated type FGM, however, has many bonded interfaces which increases a possibility to form an imperfect bonding condition.

We applied an ultrasonic image to investigate an individual layer and interface of FGM. For this purpose we prepared functionally gradient PSZ-Ni specimens of 6 layer with 100, 90, 80, 60, 40, 0% PSZ. The specimens were sintered from well mixed and laminated powder by a plasma activated sintering method by which a high density product can be sintered within a short time as compared with a conventional hot press method. The total thickness of the specimens were A; 7.99mm, B; 4.65mm and C; 2.77mm.

An internal structure of the six layers were observed by the ultrasonic image with 25MHz. The image showed almostly no defect in specimen C, however, slight defects, probably micro cracks, were recognized just after sintering in specimens A and B. during a non destructive observation of the specimen A for more than one month, the defects largely grew after 13 days then changed to crack image after 37 days. This crack can be seen by an optical image from the outside at the depth and location indicated by the ultrasonic image. These defects were not observed in specimens sintered under well controlled heating and cooling conditions referred to the ultrasonic image.

## **ULTRASONIC NON-DESTRUCTIVE TESTING OF THE DIFFERENT COMPONENTS OF THE AIRCRAFT MADE FROM THE CARBON FIBER REINFORCED PLASTICS**

R. Regazzo, M. Regazzová, J. Válková, & P. Pros, R&R NDT, Czech Republic

The article refers to an ultrasonic non-destructive testing different components of the aircrafts. These details are constituted from carbon fiber reinforced plastics (CFRP).

Different methods of ultrasonic NDT are presented. Ultrasonic testing with immersion coupling were made using ultrasonic flaw detector KB 6000 firm Krautkrämer-Branson, USA and immersion probe L5M firm Krautkrämer, Germany.

Ultrasonic testing with contact coupling were made using the newest ultrasonic flaw detector USN 50 with DGS module firm Krautkrämer and double (TR) probe SEB2KF5 firm Krautkrämer.

In this article there are presented and documented the practical results of the immersion and contact ultrasonic testing of different components of the aircrafts, which are made in the air industry in the Czech Republic.

## **STUDY OF INTERFACIAL MICROSTRUCTURE IN SiC/SiC CONTINUOUS FIBER CERAMIC COMPOSITES BY ACOUSTIC MICROSCOPY**

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Low-stress, low-temperature chemical vapor deposition infiltration (CVI) process avoids the pitfalls of conventional ceramic processing of fiber-reinforced ceramic composites. The potential of high-temperature applications of CVI composites would be enhanced by the information gained from characterization of the interfacial microstructure in various types of CVI composites, prior to and after high-temperature usage. From this point of view, we have examined three types of SiC (Nicalon) fiber/SiC matrix (ie., SiC<sub>f</sub>/SiC<sub>m</sub>) CVI composites with different fiber orientation (0-90°, 0-30°, and 0-45°), using the Ernst Leitz Scanning Acoustic Microscope (ELSAM) at 1.9 Ghz frequency, with a resolution of 1.0 micron, or better. The technique allows us to image and study the variations in the elastic properties (elastic modulus and impedance, that is, density x velocity) of the fibers, coatings, and the matrix. For these composites, surface and sub-surface areas (at depths up to 30 microns), ranging from 62.5 x 62.5 microns to 500 x 500 microns were scanned. The fringe width in the cross section of the fibers can be used to determine the Rayleigh Velocity (6.5 km/s. Thus shear velocity,  $v_s = (6.5/0.95)$  km/s = 6.737 km/s, and shear modulus = 145.2 Gpa. This value is in good agreement with the shear modulus value obtained for the SiC fiber from back-scattering Brillouin spectroscopy. High resolution acoustic micrographs of a SiC<sub>f</sub>/SiC<sub>m</sub> composites are used to

compare the elastic properties of the fiber and the matrix. Variations in the image colors are used to characterize the variations in the elastic moduli of the matrix, fiber, and coatings, as well as microstructure of SiC and in graphite speckles within the SiC layer may be interpreted to represent different processing methods.

## **IN PROCESS NDE OF COMPOSITES FOR CIVIL ENGINEERING APPLICATIONS**

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Composite materials extensively utilized in aerospace applications are finding increased use for civil engineering structures. By nature, these applications require large amounts of material and inexpensive processes. The first use of composites is in demanding applications such as bridge structures and earthquake retrofit of concrete column supports. The aerospace practices of raw material control and manufacturing are too expensive and slow for civil applications. This paper will present the requirements and development of process control methodology for civil engineering use of organic matrix composite materials. To meet requirements for process control and life-damage inspection the development study evaluates in-situ sensors and acoustical/ultrasonic methods. The test's program is developing and validating fiber optic based strain sensors, acoustical embedded sensors and guided wave ultrasonic. Using these test methods cure monitoring, material properties such as modulus and structural damage are measured over large material area. Utilization of in-situ or embedded sensors is particularly attractive because it is applicable for processing and structural lifetime damage monitoring.

## **POTENTIAL AND LIMITATIONS OF MICROWAVE NDE METHODS FOR INSPECTING GRAPHITE COMPOSITES**

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Microwave NDE methods have shown great potential for inspecting the interior of various dielectric composites. These methods are also well suited for inspecting surface features/defects such as cracks and impact damage in graphite composites. However, due to the high conductivity associated with graphite composites, the utility of microwave NDE methods for inspecting the interior these composites has been very limited. Polarization properties of microwave signals may be used to penetrate inside graphite composites depending on the orientation of the graphite fibers used to make the composite structure. Furthermore, frequency of operation may also be adjusted to provide for a reasonable depth of penetration. specially prepared graphite/epoxy composite samples with embedded materials (backing paper) at various depths are inspected using a near-field microwave NDE technique. The influence of the operating frequency and the signal polarization is investigated. Where possible, images of these "defects" are produced and presented. A discussion of the limitations associated with microwave NDE methods applied to inspecting graphite composites is also presented.

## **DAMPING IN MAGNESIUM MATRIX COMPOSITES**

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Magnesium matrix composites with short fibers and particles are potential materials for aero and space applications. The change of tensile strength of particle reinforced magnesium depends on the production method. The strength of fibre reinforced magnesium is strongly influenced by the aspect ratio of the fibre. Fibre reinforcement can also cause a decrease in damping. This is dependent on temperature and the microstructure. Damping may change also after thermomechanical treatment.

This paper presents experimental results obtained by the measurement of the logarithmic decrement and the stress relaxation.

Characterization of the internal friction is described. The paper aims to discuss different physical mechanisms controlled by the microstructure of magnesium matrix composites.

## **INTERNAL FRICTION CHARACTERIZATION OF METAL MATRIX COMPOSITES**

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In MMC's the metal matrix is exposed to plastic deformation and damage accumulation in the region close to the reinforcements, following mechanical or thermal stress. In this connection, Al-4%Cu based MMC's reinforced with 10%, 20% and 30%  $\text{Al}_2\text{O}_3$  short fibres were produced by "Squeeze Casting" and "Low Pressure Infiltration", and then characterized by internal friction (IF) measurements.

Taking IF measurements as a function of the temperature between 100 and 300 K, the existence of a maximum of damping was confirmed in the composites around 150 K during cooling at a vibration frequency of 0.05 Hz (see C Girard, PhD Thesis, INSA Lyon 1994). This maximum is absent in the corresponding unreinforced AlCu alloy, and its height is strongly dependent on the cooling rate. The maximum was therefore attributed to the generation and motion of dislocations produced by the high thermal stresses at metal-fibre interfaces.

The maximum height was found to be a reliable index to characterize and to measure the relaxation of thermal stresses at metal-fibre interfaces by the plastic deformation of the metal matrix.

Following certain thermomechanical treatments, the composites exhibit no more the IF maximum during cooling from 300 to 100 K, pointing out that the thermal stresses at metal-fibre interfaces are relaxed in some other way than the plastic deformation in the metal matrix. An analysis at the optical microscope showed that thermal stresses are in this case relaxed by cracks at metal-fibre interfaces. These cracks can be recovered by annealing the composites at an opportune temperature, and afterwards the IF maximum appears again. On this basis, IF measurements have allowed us to identify whether damage accumulation at matrix-fibre interfaces or plastic flow in the matrix take place for different composite specimens and for different conditions of thermomechanical treatments. The distribution of internal stresses in the composite can also be determined. Internal friction can "feel" both the development of cracks and plastic flow at matrix-fibre interfaces in MMC's at the very initial stage, non destructively, and averaging over a large amount of material. It can be advantageously used to characterize the quality of interfaces in MMC's.

#### **ELECTRICAL RESISTOMETRY OF Mg-BASED MICROCRYSTALLINE ALLOYS AND Mg-BASED COMPOSITES**

P. Vostry, I. Stulíková & M. Samatová, Charles University, Czech Republic; & J. Kiehn, K. Kainer, & F.M. Knoop, Technical University Clausthal, Germany

Conventionally prepared Mg-based alloys have been used widely for more than 40 years. Recently rapid solidification processing has been identified as the promising approach to achieve improvements in the properties of these alloys. The strengthening effect of ceramic fibres in the bulk Mg-based alloys matrix is investigated at the same time. This contribution deals with the results of nondestructive investigation of the thermal stability of Mg-based rapidly solidified alloys and Mg-based composites.

Specimens of the microcrystalline commercial alloy WE 54 were prepared by gas atomization and by following extrusion at 250 C or 400 C. Their thermal stability was studied by isochronal annealing from room temperature to 500 C by means of electrical resistivity measurements. The results were compared to the results obtained on conventionally prepared WE 54 alloy and to the results obtained on melt spun microcrystalline ribbons of corresponding reference binary alloys Mg-Y and Mg-Nd and were explained by precipitation effects.

Specimens of the commercial alloy QE 22 and cp-Mg were reinforced by 22 vol% Saffil short fibres, exposed to the normalization heat treatment T4 and isochronally annealed up to 300 C. The annealing response of electrical resistivity was measured and compared to the annealing response of non-reinforced materials. The results show the influence of strengthening fibres on the kinetics of precipitation processes.

## ANALYSIS OF MAJOR AND MINOR ELEMENTS IN GOLD JEWELRY BY XRF MODIFIED PROPORTIONAL FACTOR METHOD

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A series of alloy standards applied to analyze major and minor metals in gold jewelry by XRF method have been created in this paper. Theory of matrix correction is developed. the principle of proportional factor method is further modified and perfected. A new analytical method has been proposed.

Analytical Range:      Major elements: Au 40- 100%, Ag 0.05-25%, Cu 0.05-35%.  
                                 Minor elements: Zn, Fe, Ni, Sn, Pb 0.01-0.8%.

Advantages of this method: Nondestruction of sample, high-accuracy, low cost, simple and easy operation, extremely rapid and convenient etc.

The formula to calculate the analyte content ( $C_i$ ) is as follows:

$$C_i \% = \frac{A_{ij} / K_{ij}}{\sum A_{ij} / K_{ij}} \times 100 \quad (1)$$

Where  $A_{ij}$  and  $K_{ij}$  are the intensity ratios of the analytical element  $i$  and internal reference element  $j$  in sample and standard respectively.

Above formula can only apply to analyze the thin sample, when the sample is thick one, it must carry out intensity correction.

An equation of intensity correction is given by:

$$I_i^r = \sum \delta_{im} I_m + \beta_i \quad (2)$$

Where  $I_i^r$  is the correction intensity of analytical element  $i$ ,  $I_m$  is the measured intensity of matrix element  $m$ ,  $\delta_{im}$  is the influence coefficient of the matrix element  $m$  on  $i$ ,  $\beta_i$  is the constant.

The shape and area of gold jewelry are variables, so  $A_{ij}$  (or  $K_{ij}$ ) of formula (1) must carry out modification. Modification formula is as follows:

$$A_{ij} = \eta_i A'_{ij} \quad (3)$$

Where  $A_{ij} = I_i^r / K_{ij}$ ,  $A'_{ij}$  is the measured intensity ratio of the analytical element  $i$  to internal reference element  $j$  in sample,  $\eta_i$  is the modification coefficient (see other paper).

## **CRYSTAL GROWTH RATE OF POTASSIUM SULPHATE FOR COOLING CRYSTALLIZATION IN ULTRASONIC FIELD**

J. Hofmann & V. Roubik, Institute of Chemical Technology, Czech Republic

Results of experiments which are dealing with the effect of ultrasonic field on cooling crystallization from aqueous solution of potassium sulphate, are described. If a sufficient amount of solution has been provided (e.g., by mixing), the crystal growth rate is influenced especially by the incorporation of particles into the crystal lattice. Ultrasonic field has a similar effect on the growth kinetics of crystals in solution as mixing. The effect of the ultrasonic field on crystal growth rate has been studied. The crystal growth rate has been determined by the population balance analysis of crystal size distributions measured for the cooling crystallization of potassium sulphate. The steady-state crystals size distribution has been measured by a photoelectric sensor of the counter of particles.

The study of cooling crystallization with or without ultrasonic field leads to following conclusions:

1. Time of detection of measurable crystal size (40-2000  $\mu\text{m}$ ) is postponed by the effect of ultrasonic field.
2. The cumulative number of crystals is increased by the effect of ultrasonic field.
3. For crystals of size 60  $\mu\text{m}$  and larger the maximum of number of crystals is not affected by the ultrasonic field.
4. Under influence of ultrasonic field the solution retained some sort of memory. This memory became weaker with increasing time.
5. The linear crystal growth rate for smaller crystals is higher for experiments with ultrasonic field than for experiments without ultrasonic field. This result is in accord with theoretical assumptions.

## **DETERMINATION AND MONITORING OF THROUGH HOLE DIAMETERS USING ACOUSTIC DIFFRACTION**

T.P. Berndt & R.E. Green, Jr., The Johns Hopkins University, USA

A method to monitor the diameter of through holes in plates has been developed. Based on the similarity between the acoustic diffraction patterns emanating from a flat piston transducer or from a through hole placed into the far field along the beam axis of such a transducer, information about the through hole's diameter itself can be obtained. The intensity distribution within the diffracted sound field along the beam axis is characterized by a well-defined series of maxima and minima, whose locations with respect to the source of diffraction (i.e. the through hole) are related to the radius of the source and the wavelength of the incident ultrasonic waves. Thus, knowing the wavelength and the location of a specific maximum or minimum, the radius of the through hole could be calculated. In our method, a small-aperture ultrasonic detector is placed at a fixed location



along the beam axis. Then, using a novel algorithm, the frequency of a computer controlled RF signal fed into the transmitting ultrasonic transducer is automatically varied so long until the location of a minimum corresponds to that of the receiving pinducer. Once the algorithm has locked-in to this state, a continuously updated value for the frequency, together with the fixed location of the pinducer, provide a value for the current radius of the through hole. This method has the potential to detect changes of the radius in the micro- and submicrometer region. In addition, due to the differential nature of the computer algorithm the accuracy and repeatability of the calculated final values is relatively insensitive to common noise problems. Results from first experiments using 2-5mm thick plates with 1-3mm through holes in a water bath are presented. Eventually, this method shall be extended to monitor in-situ thickness changes in plated through holes in the electrolytic plating of printed wiring boards.

### **X-RAY DIFFRACTION APPLIED TO PROCESS MONITORING**

C.O. Ruud, The Pennsylvania State University, U.S.A.

The need for real-time in-situ characterization of materials during processing is being recognized increasingly. In the field of X-ray analysis the X-ray fluorescence (spectroscopy) techniques have been successfully applied to in-process inspection, while successes in X-ray diffraction have been sparse. Process monitoring X-ray diffraction (XRD) characterization techniques should be rapid, non-contacting, and tolerant of detector to workpiece distance variation. The position sensitive scintillation detector (PSSD) developed at Penn. State Univ., Ruud-Barrett PSSD, is unique in its ability to satisfy these requirements, and has been used successfully in measuring materials characteristics in real-time under manufacturing processing conditions.

XRD has been a major method for the characterization of materials for the last several decades, and can be used to identify phases, measure composition, assess grain size and crystallographic orientation, measure film thickness, determine residual stress, etc. X-ray diffractometers using the fiber optic based PSSD have been demonstrated to be applicable to in-process monitoring of several important material characteristics. Several in-process PSSD XRD applications will be described.

### **MICROWAVE DIELECTRIC CHARACTERIZATION OF LOW DENSITY GLASS FIBERS WITH RESIN BINDER**

N. Qaddoumi, S. Ganchev & R. Zoughi, Colorado State University, USA

Dielectric properties of glass fibers with varying levels of resin binder (from no resin to about 25%) are measured in the frequency range of 6 to 24 GHz. The dielectric properties of the resin are also investigated. These measurements are conducted using a well known short circuited waveguide method which is suitable for dielectric constant measurement of both liquids and fiberglass samples. The goal of this investigation is to

use the potential of dielectric characterization of low density fiberglass products to estimate the amount of resin binder used when curing. In addition, the state of cure is also investigated. This information may be used in an on-line system to determine the uniformity of curing (amount of resin binder) in low density fiberglass products. This paper presents the dielectric properties of these products as a function of microwave frequency as well as the effect of varying cure states on the dielectric properties of these glass fibers. The potential of using a dielectric characterization technique for on-line measurements will be discussed as well.

### **NONDESTRUCTIVE QUALITY AND PROCESS CONTROL IN INJECTION MOULDING POLYMER MANUFACTURE WITH MICROWAVES**

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Stuttgart, Germany

Quality control of products is closely related to the demand of their safety. As the quality of injection moulded parts depends strongly on the manufacturing process, it is important to analyze its influence on the final product.

Microwaves respond in a sensitive way on dielectric properties and on their anisotropy. Both are affected by the injection moulding process. We show that microwave anisotropy analysis can be used to measure orientation fields in short glass fibre reinforced and liquid crystal polymers. As these measurements are performed in a very short time, feedback should be possible to optimize the production process.

Such a kind of feedback requires integration of the measurement into the process. This has been done in gas injection moulding: We found that the location and extension of the bubble and defect structures can be investigated and imaged with open ended waveguide systems. For the in-situ control of the injection moulding process we will present first results of a mould integrated microwave measurement.

### **ACOUSTO-ULTRASONIC DAMAGE EVALUATION IN STEEL-BELTED RADIAL TIRES**

H. Reis, University of Illinois, USA

Underinflated (80% or less than recommended operating cold inflation pressures or runflat) radial truck tires can be subjected to steel cord fatigue damage in the upper sidewall area caused by overflexing of the tire. When the tire is being serviced, often during the retreading or the remounting and inflation steps, weakened cords may break with rupture of the upper side wall (zipper mode of failure) with potential catastrophic consequences such as loss of life.

Nondestructive evaluation of defects in steel belted radial tires has been conducted to evaluate the potential of using the acousto-ultrasonic stress-wave-factor (SWF) measurement technique to identify the zipper mode of failure and to characterize defects in the tire side wall. Different types of defects (seeded defects and defects normally found in field fatigued tires) were nondestructively tested using the acousto-ultrasonic approach. The acousto-ultrasonic results from the damaged regions were compared with the results from regions without failed steel cords. Results show that the acousto-ultrasonic approach has the potential of being used in quality assurance of tires, that it is production-oriented, and that it is capable of 100% inspection of the tire casings prior to retreading.

### **MATERIALS CHARACTERIZATION OF POWDER METALLURGY PRODUCTS USING ACOUSTO-ULTRASONICS**

G.L. Workman & J.L. Walker, University of Alabama, USA

The use of acousto-ultrasonic techniques for the materials characterization of powder metallurgy (PM) parts is being investigated. The experiments reported here on small PM impact cages provides an approach for a broad range of PM components. Broad band piezoelectric transducers are used to transmit and receive the ultrasonic energy in and out of the part. Preliminary experiments on a small sample set showed that the integrated energy content of broad band ultrasonic signals in PM parts were sensitive to the presence of defects and other variations in structural integrity. Preliminary data taken before and after fabricating known defects into half of a six sample set was used to train a neural network to discriminate between the two types of parts. Analysis of these results showed that the neural networks were able to provide acceptable accept and reject criteria.

A larger sample set is currently being used to confirm the results from the earlier work. In addition to the use of neural networks for accept and reject decisions, the use of multivariate analysis techniques is being used to further resolve other levels of variations in the structural characteristics of the parts. The work in process also includes the analysis of the frequency distributions contained in the broadband signals to provide information about the type of defect or structural anomalies present in the PM part.

### **PROCESS INTEGRATED NONDESTRUCTIVE TESTING OF LASER-HARDENED COMPONENTS**

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Surface modification of components by laser beam hardening is of increasing interest because of the possibility of local treatments and its easy integration into the production line. Because of various parameters influencing the hardening process, non-destructive testing techniques are desired for monitoring the machining quality.

Up to now only techniques like metallographic inspection, x-ray diffraction and hardness measurements are being used to determine hardness, hardening depth and residual stresses. Using these techniques a statistical process control is performed in most cases. However these techniques cannot be integrated into the production line because their testing speed is too slow and most of these techniques are destructive in nature.

Micromagnetic techniques developed during the last years are found to be most appropriate for nondestructive evaluation of the machining quality of the laser hardened components. Combining the complementary information contents of the various microstructure and stress sensitive measuring parameters, a fast estimation of relevant quality parameters can be simultaneously obtained. Using a multiple regression analysis, approximation functions are obtained to determine the hardness, hardening depth and residual stresses.

Nondestructive evaluation of process/quality parameters is demonstrated for laser-hardened components using new multiparameter sensor systems. Based on these results, a prototype of a process integrated nondestructive testing system (PINT) for 100%-inspection of the laser-hardening quality is installed. Such testing devices can be applied to evaluate the components surfaces hardened through any other processes and also to evaluate the machined surfaces.

## **NONDESTRUCTIVE CHARACTERIZATION OF CURE ENHANCEMENT BY HIGH POWER ULTRASOUND OF CARBON EPOXY COMPOSITES**

T.M. Whitney & R.E. Green, Jr., The Johns Hopkins University, USA

High power ultrasound is used to enhance the cure of AS4/3501-6 carbon epoxy composite. Composite panels are insonified through the caul plate, by a high power ultrasonic horn, while curing. Enhancements in the properties of the cured composite panels, relative to panels cured in the same global environment (*sans* insonification), will be presented. Enhancements of the cure process, relative to panels which were not insonified, will also be presented.

A novel method is applied to the monitoring of the degree of cure. The resonance spectrum of a steel caul plate, on which the composite panel to be cured is vacuum bagged, is measured periodically as the temperature is raised to the cure temperature and during the subsequent cure and cool-down periods. Because the curing composite acts to change the boundary conditions, the resonance spectrum changes as the composite cures. Differences in amplitude, frequency, and damping are related to the degree of cure of the composite at each temperature.

## **DIFFRACTION TECHNIQUES IN ENGINEERING APPLICATIONS**

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Oak Ridge National Laboratory, USA

Diffraction techniques applied to crystalline materials provide quantitative information about the crystallographic structure and mechanical condition of the material. Those two characteristics influence the chemical, physical, mechanical, and technological properties of a component. A concerted application of neutron and x-ray diffraction allows one to study comprehensively the bulk and subsurface variations of such material characteristics as crystallographic texture, residual stress, and cold work. The Residual Stress User Center at the Oak Ridge National Laboratory offers to the academic and industrial researchers both the neutron and x-ray diffraction capabilities. Examples of the application of those capabilities to thin film, metal, ceramic and composite material technologies will be presented.

## **MATERIALS CHARACTERIZATION WITH COLD NEUTRONS**

H.J. Prask, National Institute of Standards and Technology, USA

NIST has established at its Gaithersburg site the nation's first dedicated facility for "cold neutron" research (the CNRF). This new national facility is providing researchers in such fields as materials science, physics, chemistry, and biology state-of-the-art instrumentation which takes advantage of the unique properties of cold neutrons. As of December 1994, eleven of the planned fifteen new instruments were operations. One-quarter to two-thirds of total beam time is available to the general research community, at no charge, through a proposal evaluation system.

The main focus of this paper will be to review the principal techniques in which cold neutrons are currently used at the CNRF for nondestructive materials characterization: small-angle neutron scattering (SANS), neutron reflectometry, neutron depth profiling (NDP), and prompt-gamma activation analysis (PGAA). Recent examples of representative work in each of these areas will be presented.

## **NEUTRON DEPOLARIZATION ANALYSIS AT PULSED NEUTRON SOURCES FOR TESTING OF MICROMAGNETIC STRUCTURE AND RESIDUAL STRESSES OF MAGNETIC LAYERS**

L.P. Chernenko & D.A. Korneev, Frank Laboratory of Neutron Physics, Russia; & J. Schreiber & W. Theiner, Fraunhofer-Institute for Nondestructive Testing, Germany

Analyzing the depolarization rate of neutrons transmitted through magnetically disordered samples allows to characterize functional films and coatings up to 1000 nm thickness on the basis of their magnetic properties. It will be shown that the magnetic

order/disorder and special non-homogeneities within the magnetic layers can be detected, the stress states can be tested by magnetoelastic effects and a quantitative estimation of the magnetic quantities within the layers can be given, i. e. saturation and remanent magnetization along the film plane, coercive field strength, averaged size of magnetic ordered regions or domains and content of nonmagnetic inclusions in the magnetic layer. The time of flight method of the neutron depolarization measurements has the advantage that the wave length dependence of the depolarization function can be investigated. A pronounced minimum in this function points at correlated magnetic order perpendicular to the field and polarization direction. These rather magnetically ordered regions are in general induced by compressive (tensile) stresses.

First results of nondestructive evaluation of Ni-dispersion layers on Al cylinders are discussed by the neutron depolarization analysis and the results are compared with that of other ndt-methods.

### **NONDESTRUCTIVE MORPHOLOGICAL CHARACTERIZATION OF LATENT AND ETCHED ION TRACKS IN PETP BY SANS**

F. Haeussler, M. Hempel, M. Kröning & H. Baumbach, Fraunhofer-Institut fuer zerstöerungsfreie Prüfverfahren; & W. Birkholz, Umweltministerium des Landes Mecklenburg-Vorpommern, Germany

Solid state nuclear track detectors (SSNTD) represent passive detectors for ionizing radiation and can be used for the registration and identification of charged particles. The production of nuclear filters with defined porous radii in the  $\mu$ -level is achievable with special etching procedures of SSNTD [1,2]. The non-destructive characterization of the morphology of the latent tracks in SSNTD is of high importance. In the last years several studies on irradiated SSNTD-materials were published. First experiments to get information about latent ion tracks by small-angle neutron scattering (SANS) are discussed by Albrecht and co-workers [1]. Furthermore, the study of the first steps of the etching procedure of latent ion tracks gives some knowledge for the optimization of both the technique of irradiation and subsequent etching and the properties of polymer material of SSNTD.

In this contribution the study of various latent ion tracks in SSNTD by SANS [3] before and after etching shall be presented. The experiments were realized on the MURN facility of the pulsed reactor IBR-2 at the JINR, Dubna. A stack of several square shaped PETP foils (the thickness of a single foil is of about 10  $\mu$ m, total thickness of the stack is of about 0.8 mm) are used for the measurements. At sample position the neutron beam cross section is of about 1.5 cm<sup>2</sup>. The PETP samples irradiated by Kr-84 (energy 2.50 MeV per nucleus) were investigated. For studying both the irradiated latent tracks and the etched tracks, a low track density of about  $1 \cdot 10^9$  cm<sup>-2</sup> was used [4].

The slope of the scattering curve of irradiated and etched ion tracks using the Guinier plot indicates the size range of the radial extension of the latent ion track and the etched track. The radii of gyration  $R_g$  calculated from the slope of the scattering curve are of about 7 nm for latent ion tracks and move within a range of about 5 nm to 15 nm for etched tracks. The  $R_g$  values are increasing with the etching time. The Guinier-plots of the etched samples show regions with different slopes, i.e. two  $R_g$  values. The  $R_g$  values measured by SANS were compared with the  $R_e$  values measured by a conductometric method. Comparing the SANS pattern with some modelled scattering curves calculated from various track structure models, the experimental results are discussed more in details.

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#### **CHARACTERIZATION OF MICROSTRUCTURE OF PLASTICALLY DEFORMED AND THERMICALLY TREATED CARBON STEEL BY MEANS OF POSITRON ANNIHILATION LIFE TIME SPECTROSCOPY IN COMPARISON WITH MICROMAGNETIC METHODS**

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The positron annihilation technique provides information to the concentration of crystal lattice defects. Analyzing life time spectra, vacancy concentration and dislocation densities can be determined separately. Information about vacancy clusters (microvoids) cannot be obtained by any other method with the same sensitivity.

The results of recovery experiments on plastically deformed nickel, iron and carbon steels will be presented. By comparing the results of positron annihilation and micromagnetics important information on annealing of crystal lattice defects can be obtained. In comparison with micromagnetics positron annihilation is relatively insensitive to the chemical composition of the material. Therefore calibration is less problematic.

The amplitude of Barkhausen noise and then mean life time of positrons behave in the same manner as a function of temperature if plastically deformed material is annealed during time linear heating. The separation of positron life time spectra into different time components can give additional information about interaction with point defects, especially concerning the formation and dissolution of vacancy clusters. Annealing effects due to vacancies and dislocations can also be separated.

## POSITRON ANNIHILATION MEASUREMENTS IN Zr AT HIGH TEMPERATURES

R. Král & V. Gröger & G. Krexner, University of Vienna, Austria

The temperature dependence of the positron lifetime and Doppler-broadening of the positron-annihilation  $\gamma$  line has been measured in Zr in the temperature range  $20 < T < 1700$  C with the aim to investigate the formation of equilibrium thermal vacancies. For the experiments a narrow high-vacuum furnace (outer diameter 30mm) with graphite tube-shaped heating element and sealed-source specimen (electron beam welding) was used. The specimen was supported on its position in the axis of the heating tube by massive W-Ta thermocouple rods. Although a rather high vacancy concentration is to be expected according to the comparison of X-ray and dilatometric measurements, only weak indications of effects ascribable to the generation of equilibrium vacancies were observed (at temperatures above 1300 C). This result is in accordance with the earlier Doppler-broadening experiments. Both lifetime and S-parameter show a predominantly linear increase below the h.c.p.  $\rightarrow$  b.c.c. transformation temperature  $T_{\alpha-\beta}$ . No discontinuous change of experimental data was observed at the phase transformation, although lower values of lifetime and S-parameter should follow from the negative volume change. The phase transformation is discussed in terms of vacancy and defect formation and its effect on positron annihilation, and experimental data are compared to theoretical predictions. For  $T_{\alpha-\beta} < T < 1300$  C, a second linear, but more pronounced, dependence is observed. For the temperatures above 1300 C there is some tendency towards saturation behavior of S-parameter and lifetime data which can be associated with vacancy trapping. However, the effect is very weak and within the limitations of experimental data not well established. fitting of single trapping model to the experimental data gives the maximum intensity from trapped positrons to be about 2% for 1700 C. Effects of impurities, especially solute gases, and possible influence of fast jumps of atoms into vacant positions (connected with anomalous diffusion) on the vacancy trapping is discussed.



## **DISLOCATION DENSITY MEASUREMENT AND POSITRON ANNIHILATION**

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There exist only a few promising experimental results concerning a marked influence of Burgers vector and jog density (but unfortunately as well impurity content) in certain dislocated fcc metals on the positron annihilation parameters, and concerning different influences of edge and screw dislocations in iron. We aim to obtain a more general picture of the positron annihilation process at dislocations in metals for establishing positron annihilation spectroscopy as method for the measurement of global dislocation densities. We study unjogged edge dislocations at interphase boundaries (misfit dislocations as well as structural misfit dislocations of very small Burgers vector being associated with ledges) confirming the possibility to measure average absolute values of Burgers vectors in simple cases. We investigate fcc single crystals deformed in single and multiple glide, respectively, to clarify the role of jog densities in positron annihilation at dislocations. Specific deformation sequences at low temperatures and elevated temperatures of bcc single crystals are performed for obtaining different proportions of edge and screw dislocations and to study possible differences in positron annihilation parameters. Deformation induced point defects are carefully annealed out and electrical resistivity measurements are used in combination with a TEM investigation for obtaining the dislocation densities independently. High resolution measurements of positron lifetimes and Doppler broadening are performed at room temperature. The implications of the results for dislocation density measurement are discussed.

## **ELECTRON DIFFRACTION STUDY OF LANGMUIR-BLODGETT LIPID FILMS**

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Among structural methods electron diffraction is one of the most convenient and informative. Due to the strong interaction of the electron beam and the sample materials high-quality diffraction patterns can be obtained from rather thin films. A new material for constructing sensing devices is thin films made by the Langmuir-Blodgett (LB) technique from organic molecules. This method permits fabrication of multilayer films with control of different film parameters and is of interest for purposes of bioelectronics. Electron diffraction can be used for control of LB films during preparation. The high-resolution technique for investigation of LB films structure is the purpose of this work. Structure of films made from complex organic molecules may be changed by electron beams or vacuum. Information about the structural changes can be obtained by precision intensity profile analysis of electron diffraction. We constructed a high-precision system for electron diffraction intensity measurement. Two-dimensional electron diffraction patterns can be

measured with an accuracy of about 1% in intensity. Short time registration of the diffraction pattern allows analysis of the structure of short lived samples in real-time. Structural investigation of lipid Langmuir-Blodgett films was been made with this technique. A molecular model was proposed.

### **NONDESTRUCTIVE MATERIALS CHARACTERIZATION FOR ARCHITECTURAL CONSERVATION**

R.A. Livingston, Federal Highway Administration, USA

Nondestructive methods for materials characterization can be applied to a wide range of problems in the conservation of cultural monuments and historic structures. Since destructive testing is generally prohibited in this field, the nondestructive approach is extremely important. NDT can be used in architectural archaeology to determine provenance of materials, dates of construction or changes in technology. For analysis of structural conditions, NDT tomographic methods can be used to look below frescoes, mosaics or other surface layers to identify materials in terms of physical properties such as density or modulus of elasticity. Materials characterization in terms of chemical or mineralogical composition is also required. NDT characterization of the deterioration of materials plays an important role in diagnostics for architectural conservation. This kind of characterization can involve changes in the material itself, such as corrosion of metals, or the detection of harmful environmental factors such as water or chlorides.

### **DAMPING OF CONCRETE BEAMS; PLAIN, REINFORCED AND PRESTRESSED**

R. Kohoutek, University of Wollongong, Australia

A forced vibration technique for the measurement of elastic modulus and loss factor of concrete beams, which includes resonant frequencies, is used to determine damping properties. Several different beams made of plain concrete, reinforced concrete, and prestressed concrete have been cast and for a constant initial deflections exciting forces and phase shifts measured. The results indicate large differences of damping between resonant and non-resonant regions, but also considerable damping dependency on the concrete mix, reinforcing ratio, and prestress force. The damping dependency on frequency and mix design could be used for a passive control of vibrations for structures. Two different forms of excitations were used, flexural and axial, which develop different stress fields in the same beam. Furthermore, the method, which is exact in terms of the governing differential equation, can be also used to testing other materials.

## HOW CALCAREOUS LAYERS AFFECT ULTRASONIC THICKNESS GAGING

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Underwater structures--such as off-shores, immersed pipelines, etc.--require periodic thickness measurement of the metallic walls in order to monitor the loss of material due to corrosion. A typical problem which affects ultrasonic thickness gaging of such structures is caused by the encrustation, mainly of biological type, that covers the surfaces exposed to the sea water. This requires a preliminary cleaning of the parts, which must be performed by the operator before the inspection; furthermore it hinders the automatic execution of the inspection by means of small remotely-guided submarines.

The aim of this work is to explore the feasibility of thickness gaging of steel walls (in the typical thickness range of off-shore structures, i.e. some tens of mm) if the encrusting layer is not removed. The research has been performed considering specimens covered by simulated encrustation. The specimens are formed by carbon steel plates, machined in stepwise shape to obtain thickness level ranging from 12 to 20 mm with steps of 1 or 2 mm; the artificial encrustation has been formed using two kinds of calcareous materials, namely lime and hydraulic lime, in layers ranging from 1 to 5 mm in steps of 1 mm. All possible combinations of thickness values for plate and encrustation have been considered.

Ultrasonic measurement has been performed in immersion, adopting the pulse-echo technique and using transducers with nominal frequency of 1 and 2 MHz; higher frequency values, which could be desirable to achieve better thickness resolution, are not suitable as the signal is completely canceled by the calcareous layer. The experiments show that the frequency value of 1 MHz allows for satisfactory measurement over all considered cases, when the frequency value is 2 MHz the measurement is possible only for encrustation not thicker than 1 mm.

This behavior can be justified considering the frequency spectrum of the reflected echoes, obtained by applying the Fourier transform of the signal. It has been found that as far as the 1 MHz transducer is concerned the spectrum remains centered about the nominal frequency, while in the case of the 2 MHz (or more) transducer the spectrum is spread over a large frequency range and the pulse loses its shape in the time domain.

These results suggest that, adopting proper values of test frequency and *ad hoc* procedures, ultrasonic thickness gaging of encrusted walls is possible.

## **INVESTIGATION OF SPURIOUS ECHOES RECEIVED IN AN ULTRASONIC INSPECTION OF AN OIL FIELD TOOL**

D.E. Bray, W. Tang, B. Bidigare, & L.R. Cornwell, Texas A&M University, USA

Ultrasonic 45° shear wave inspections of oil field tools showed significant indications at locations midway along the length. The tools are 4140 steel and approximately 152 mm in diameter and 2 m long. A 58 mm hole is drilled the length of the bar. At various stages in the manufacturing process, the tool is rotary straightened, gun-drilled, quenched and tempered, stress relieved, milled and machined. Following confirmation of the indications obtained by the inspector, an sectioning process was begun to identify the source of the reflections. Starting first with a section approximately 75x125 mm and 40 mm thick cut from the bar, incremental layers were removed and the new surface investigated by numerous techniques, including wet magnetics, radiography, eddy current and metallography. At no time were any sources of the reflections identified. The cutting was stopped, with a remaining smaller piece now 9 mm thick. In the various steps of the investigation, an increase in attenuation toward the center of the section became apparent. Where attenuation values of 20 to 23 dB/m were found with 45° shear waves at the ends of the bar, shear wave values across the thickness in the small sample ranged from 127 to 165 dB/m. A programmed stress relief performed on part of the small section failed to change the attenuation. Further investigations using X-ray diffraction are planned to help identify the source both of the original indications and of the high attenuation found in the mid section of the bar.

## **THE EVALUATION OF INTEGRITY OF CERAMIC-METAL JOINTS AND CERAMIC COATINGS BY C-MODE ACOUSTIC MICROSCOPY**

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The structural integrity of alumina - Fe-Ni-Co - alumina joints bonded with an active braze Ag-Cu-Ti have been evaluated by using a reflection type acoustic microscope operating at frequencies up to 100 MHz. From the amplitude based C-scan images of the samples the software calculates the percentage of the area where certain amplitude threshold has been exceeded. This percentage has been used as a quantitative measure for the quality of the joint. The correlation between the measured unbonded areas and the corresponding shear strength of the joint was experimentally verified. For detection of defects in brazed  $\text{Al}_2\text{O}_3/\text{Al}_2\text{O}_3$ - joints with a Fe-Ni-Co insert metal a focussed high-frequency transducer was used. The measurements were made from both sides of the joint, thus enabling each ceramic/braze interface to be evaluated separately.

The same ultrasonic technique has also been applied to vacuum brazed-silicon nitride-metal joints developed for service temperatures exceeding 1000 C. A clear correspondence could be detected between the C-SAM images, joint strengths and fracture surfaces.

For the evaluation of the porosity and elastic properties of thick thermally sprayed alumina coatings the velocity of Rayleigh-waves in the coating has been measured. The measurements have been carried out by using the conventional  $V(z)$ -technique in acoustic microscopy or the velocity has been determined by measurement of angles of total reflection. To simplify this measurement special transducers have been developed. Based on the sound velocities measured the elastic modulus and Poisson ratio for the coating material has been calculated. The coating thickness has typically been several hundred microns but by increasing the ultrasonic frequency also thinner coatings can be characterized.

### **ULTRASONIC CHARACTERIZATION OF DEFECTS IN LEAD-MAGNESIUM NIOBATE (PMN) SMART MATERIALS**

J.R. Bernstein, J.W. Wagner and J.B. Spicer, The Johns Hopkins University, USA

Materials and structures composed of the electrostrictive ceramic lead-magnesium niobate (PMN) are currently being produced for smart materials applications. Electrostrictive materials such as PMN are known to exhibit desirable electromechanical properties for applications where low hysteresis between an applied field and the material deformation is required. As occurs in many ceramic materials systems, the processing of PMN may induce various defects in the material which may adversely affect the performance of the material.

In this work, a variety of ultrasonic, nondestructive techniques have been used to characterize the degradation mechanisms of PMN-based devices. The defects investigated included voids intentionally introduced during the manufacturing process and cracks resulting from extended/accelerated electromechanical cycling of the devices. This work shows that traditional pulse-echo and through-transmission ultrasonic testing modes have proven useful in locating gross defects such as voids and through cracks. Ultrasonic second-harmonic generation has been used to help identify and quantify the extent of distributed microcracking in the ceramic as it is cycled. By performing these ultrasonic tests as a function of an external applied bias voltage to the PMN, additional information about the defect content of the material has been obtained.

## **MEASUREMENT OF ADHESION STRENGTH USING NONLINEAR ACOUSTICS**

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Weak adhesion strength or delaminations can be detected qualitatively by conventional techniques such as the determination of the interface reflection coefficient of an ultrasonic or thermal wave. However, a quantitative determination of adhesion strength is difficult and not practical for industrial applications. We have developed a new method using nonlinear acoustics allowing the determination of the adhesion strength of simply bonded or coated structures, as well as of more complex structures like fibre matrix composites. The adhesion strength of these joined materials is dominantly described by the behavior of their interfaces, usually the weakest parts of the composite. Using ultrasonic waves of sufficient amplitude, the transmitted ultrasonic wave is modulated by the nonlinear elastic behavior of the interface. Depending on the quality of the adhesion strength higher harmonics of the fundamental frequency are generated. Theoretical calculations give a correlation between the amplitude of the measured higher harmonics and the adhesion strength of the composite. Therefore, evaluating the amplitude of the generated higher harmonics, the adhesion strength of the composites can be determined nondestructively. Using this method, measurements of the adhesion strength at the components can be realized which might be helpful for fatigue life predictions of the investigated components. The potential of this new technique will be demonstrated on several joint materials, especially on mechanical loaded CFC (carbon fibers in carbon matrix).

## **ON-LINE ULTRASONIC TESTING SYSTEM OF THE NEXT GENERATION BY USING REAL-TIME CHIRP PULSE COMPRESSION PROCESSING**

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In the ultrasonic testing of material, higher frequency components of the echo signal have more precise information that may distinguish types of flaws or may characterize material. However, the applications of the ultrasonic testing employing the high frequency and wide band signal was restricted due to the heavy attenuation of the ultrasonic signal in the material, the low efficiency of the ultrasonic probe, as well as the harmful influence of the noise in the wide frequency range. Therefore, in the conventional ultrasonic testing system, relatively low frequency and narrow band signals have been actually employed for the quality guarantee of steel products to obtain the better signal-to-noise ratio (S/N) rather than to obtain more detail information from the echo signature.

To establish the high frequency and wide band ultrasonic testing technology, the pulse compression technology such as chirp signal, which is a kind of frequency modulated burst waves, is supposed to become a promising key technique in that S/N can

be dramatically improved without any sacrifice of the resolution even in the ultrasonic testing, waveform deformation due to the propagation in material should be taken into account to obtain the effective pulse compression.

In this paper, it is reported that the optimum modification of the chirp pulse waveform depending on the factors of the ultrasonic propagation in material and a real-time processing system for the pulse compression have been successfully developed. As a result of this pulse compression, high performance on both of the S/N and the resolution for flaw detection by the frequency optimization operation of the ultrasonic testing have been achieved. It is concluded that as a methodology of the next generation for the on-line flaw detection system in the steel production line, a robust inspection and evaluation technology based on the flaw and material characterization has been established.

### **AN AUTOMATIC ULTRASONIC TESTING SYSTEM FOR THE BUTT WELD ZONE OF THE GAS PIPE LINE**

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In Japan the construction of gas pipe line is required to popularize the Liquid Natural Gas (LNG). In order to reduce the cost of the construction of the gas pipe line, it is strongly required to increase the reliability of NDT and to shorten its testing time. For this purpose the development of an automatic ultrasonic testing system for the weld is very important. On the other hand, the steel plate for the gas pipe is produced by the thermo-mechanical controlled process to increase its low temperature toughness. This process results in its very strong acoustic anisotropy, and reduces the spatial resolution of the defects in the NDT.

An automatic ultrasonic testing system for the butt weld zone of the gas pipe line has been developed. This system is a multiprobe system and has two outstanding features. One is the newly developed advanced V path technique to measure the wave velocities and another is the discrimination of the spurious echo. In the anisotropic materials, the wave velocities depend on the angle of incidence. In this system the distributions of the wave velocities are measured by using the beam width with one pair of probes, after that the other probe's wave velocities are estimated. This method is very powerful especially in the multiprobe system such as using the probes which have different angle of refraction. Due to the discrimination of spurious echo in the preliminary testing, the number of measurement points for the precise testing are reduced and the testing time in case of the 750A pipe is less than twenty minutes.

## THE ULTRASONIC TESTING OF WELDING IN PLASTICS

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The methods, which now used for testing of welding joints in plastics, are based on the application of longitudinal waves with frequency 1-2 MHz. Usually, transducer of non large diameter ( $\sim 10^{-2}$ m) radiates the ultrasonic pulse in direction closed to the angle  $\beta/2$  to the surface of joint. But the main dangerous defects in products, manufactured by flash welding of plastics, are the joints of surfaces without materials diffusion, the defects which named "mirror."

In longitudinal wave, which transmits through the welding joint under the angle  $\beta$  to its surface the normal displacement  $u_n = u_l \sin \beta$  and the tangential displacement  $u_t = u_l \cos \beta$ ,  $u_l$  - displacement in directional of wave distribution. Because, the sound speed in plastics (for example polyethylene) is approximately 2000-2300 m/s, the transducer for its testing must have the large angle of prisme ( $>60^\circ$ ). Since, the  $u_n$  more large than  $u_t$ , the longitudinal wave most not reflect from the defect "mirror." For identification of this type of defects the wave which has the displacement  $u_t > u_n$ , must be used. Usually, the transverse waves are applicated for this purpose. But, at first, the plastics have a great acoustic absorption, at second, the excitation of transverse waves is difficult, because of, the sound speed is small.

For solving of this task, the method, which based on the using of two transducers, is proposed. In this method, the longitudinal acoustic waves are excited in the region of weld under the angle closed to  $n/2$ , on one plane and near 0 in other plane. The  $u_t$ , in this case more large than  $u_n$ . The testing may be released as echo- as shadow- method. The experiments have been made on the polyetilen tubes welding joints. The sensitivity of proposed method is approximately 6 dB on the  $2 \cdot 10^{-6}$  m<sup>2</sup> for echo-method and 4 dB for shadow method. The science base of proposed method and its practical realization will be describe in suggested paper.

## DETERMINATION OF THE ELASTIC CONSTANTS OF ANISOTROPIC SOLIDS WITH AN ARTIFICIAL NEURAL NETWORK

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The elastic constants  $C_{ij}$  of transversely isotropic composites and other materials are determined from ultrasonic group velocity data by an estimator-feedforward neural network (FFNN). Elastodynamic theory is used to calculate from known  $C_{ij}$ 's, the group velocities of thousands of virtual materials possessing the proper symmetry. The results of this forward problem form the training and validation sets for the FFNN, which establishes the mapping between the group velocities and the elastic moduli of a test specimen. Results



demonstrate the successful inversion of group velocity data for a single crystal specimen of zinc and several thermoplastic composite specimens.

### **NEGATIVE ELASTIC CONSTANTS IN INTERMEDIATE VALENT $\text{Sm}_x\text{La}_{1-x}\text{S}$**

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At normal pressure SmS by itself is a semiconductor. It is of some interest, because of its intermediate valent behavior at a pressure higher than 6.5 kbar. On the other hand SmS doped with 25% La becomes intermediate valent already at normal pressure and room temperature. The doping La-atoms create new occupied electronic states in the SmS-gap and this leads to the typical mixing of the  $4f$  and the  $4f-1-5d$  states.

A strong evidence of intermediate valence is a negative Poisson's ratio. Using high resolution Brillouin-scattering at a wavelength of 514 nm we measured the phase velocity of the surface acoustic waves in the (001)-plane of  $\text{Sm}_x\text{La}_{1-x}\text{S}$  ( $x \in \{1.00, 0.90, 0.75, 0.65\}$ ) at an angle of incidence of  $70^\circ$ . Applying the Levenberg-Marquard algorithm we calculated all three elastic constants ( $C_{11}$ ,  $C_{12}$  and  $C_{44}$ ) from the angular dispersion relation (on varying the direction from [100] to [110]). To achieve better results the compression-moduli have been determined, too, and linked to the elastic constants  $C_{11}$  and  $C_{12}$  in the fit-procedure.

The calculated Poisson's ratio of  $\text{Sm}_x\text{La}_{1-x}\text{S}$  shows a strong non-linear behavior as function of  $x$ . The ratio turns to be negative at a doping level below  $x=0.90$  and becomes positive again for  $\text{Sm}_{0.75}\text{La}_{0.25}\text{S}$ . The minimum is located around  $x=0.75$ . These results are in good agreement with high pressure studies of the resistivity. They also show strongest intermediate valence at  $x=0.75$ .

### **ESTIMATION OF PARAMETRIC MODELS FOR DOUBLE TRANSMISSION EXPERIMENTS ON A VISCOELASTIC PLATE**

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In this paper, a system identification approach is proposed for the determination of properties of viscoelastic plates immersed in water. A double transmission (DT) configuration [1, 2] is used for the experiments, which facilitates the calibration of measurement setup. With the DT configuration, parametric models are established for ultrasonic wave propagation through a viscoelastic plate at both normal and oblique incidence. The DT models for obliquely incident experiments are simplified by restraining the incident angle exceeding the critical angle for longitudinal waves. The absorption-dispersion properties of materials are expressed using a rational transfer function model [3]. The effects of beam spreading due to finite dimensions of transducers are taken into

account by introducing a diffraction factor into the wave propagation models [4]. A maximum likelihood estimator [5] is developed for the estimation of the model parameters. From the estimated parameters, the properties of viscoelastic plates, such as longitudinal and shear velocities, density, and thickness, are deduced, and the absorption and dispersion functions of materials for both shear and longitudinal waves are obtained. Furthermore, utilizing the estimated models, the prediction of the DT measurement can be made at different incident angles. The predicted and measured transfer functions are then compared to validate the estimates. The method will be illustrated experimentally for different viscoelastic materials.

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## **STRATEGY TOWARDS NONDESTRUCTIVE EVALUATION OF MECHANICAL PROPERTIES OF STEEL COMPONENTS**

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The most widely used acceptance specifications of materials are based on their mechanical properties. They are usually evaluated by destructive sampling. The applied procedure will satisfy the needs for quality control of the manufactured material, but most often it is not acceptable for monitoring the change in the mechanical properties associated with the microstructural changes taking place in a component in service or for process integrated evaluation of these parameters. A nondestructive way to evaluate mechanical properties is a challenge of significant economical perspective.

Direct evaluation of most mechanical properties is difficult accessible using nondestructive techniques although both nondestructive quantities and mechanical properties are determined by more or less the same microstructural features. The various complex interactions between the microstructural parameters on the one hand and nondestructive quantities and mechanical properties on the other hand have been identified

as the reason for the unsatisfactory state of nondestructive approaches.

For this reason, an extended study using more than 70 steel samples with different but known mechanical properties has been performed. Seven magnetic and magneto-elastic quantities have been measured. Using multiparametric regression algorithms, convincing correlations could be established between these quantities and strength parameters, to toughness and hardness. Use of ultrasonic quantities like velocity, scattering coefficient and acousto-elastic constants are still under evaluation. A first result obtained is a correlation between the acousto-elastic constant and the dislocation density.

The strategy towards nondestructive evaluation of mechanical properties of steel components comprises:

- fundamental investigation of the influence of individual microstructural parameters on the quantities measured,

- multiparametric evaluation using advanced regression algorithms,

- availability of robust sensors to measure different quantities simultaneously.

The contribution presents some of our experimental results and discusses our approach for the process integrated evaluation of mechanical properties.

## **INTERNAL FRICTION IN MAGNESIUM ALLOYS PREPARED BY RAPID SOLIDIFICATION**

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Magnesium is one of High-Damping Metals. This property makes it an attractive alternative to aluminium for the designer of structures. On the other hand, magnesium alloys prepared by conventional ingot methods exhibit low values of the tensile strength. Rapid solidification processing of magnesium alloys can lead to the improved mechanical strength and corrosion resistance. This is due to the production of fine microstructure, extended solid solubility and the production of new phases.

This paper describes characterization of the mechanical damping properties of Mg and a serie of Mg alloys prepared by rapid solidification. The damping mechanisms depend on the stress-induced movement of defects in the metal. In our experiments we used measurements of the logarithmic decrement as the characteristic parameter which describes the dumping properties of materials.

The strain dependence of the logarithmic decrement exhibits two regions. In the first region the logarithmic decrement is independent of the maximum strain of the bending vibration. In the second region the logarithmic decrement depends on strain. This paper

describes the physical basis of internal friction.

## **QUALIFYING INDENTATION FRACTURE TOUGHNESS TESTING BY ULTRASONICS**

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Indentation fracture toughness testing is based on the development of steady state crack configurations due to indentation of a hard indenter of special shape (sphere, Vickers pyramid) into the surface of a material and the utilization of the characteristic crack dimensions in order to estimate the materials fracture toughness. This method is particularly attractive for the characterization of new brittle materials because only small simply shaped test pieces are required. On the other hand, the method is full of unsolved problems: There is a variety of empirical indentation toughness equations contrasted with the lack of a rigorous theoretical basis on the one hand and with the lack of any standard testing procedure on the other hand. Furthermore, it is not easy to obtain information on subsurface features of the crack configurations in opaque materials. The conventional technique is the time-consuming preparation of sections through the indentation (section-and-etch technique). For several reasons it may be important to get the relevant information nondestructively either during or after indentation.

The present paper deals with the nondestructive characterization of indentation crack configurations by means of ultrasonic methods. Relevant problems and existing approaches including previous work of the authors using acoustic microscopy and high frequency acoustic emission are shortly reviewed. Current work is focused on the measurement of the characteristic dimension of Hertzian conus cracks, the distinction between the radial-Palmqvist and radial-halfpenny crack configurations, and the measurement of the near indentation delamination area in coated materials. An ultrasonic C-scan system is applied in addition to scanning acoustic microscopy. As a major result the quality of a conus crack (i.e. deviations from the ideal conus crack system) has been characterized and the diameter has been estimated on the basis of C scans performed from the backface of a slide glass specimen. The results are compared with optical images.

## **NONLINEAR ACOUSTIC PARAMETER AND STRENGTH OF SOLIDS**

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When an acoustic plane longitudinal wave propagates in solid, the waveform will be distorted due to nonlinearity of material and harmonics will be generated. A parameter  $\beta$  is used to characterize material nonlinearity. It also became a new parameter for material characterization in NDE and is increasingly important for description of material properties such as the strength of solids. Up to now,  $\beta$  is usually determined by measuring fundamental and second harmonic amplitudes using burst technique in progressive wave

field. This method can't be used for samples with thickness less than acoustic wavelength, such as the rock sample which is not too large but measured in low frequency due to its high attenuation in high frequency, or for thin film sample which needs very high frequency burst signal with very expensive equipment. The authors have studied the one dimensional nonlinear acoustic longitudinal standing wave field in a rod and a new standing wave method for determining  $\beta$  was developed, in which an acoustic impedance  $Z$  at the interface of transducer and sample was introduced. In this paper, the nonlinear standing wave field in a rod and how to measure  $Z$  and then  $\beta$  are introduced, and the relation between nonlinear acoustic parameter and strength of solid is discussed.

### **NEW DIGITAL TECHNIQUES FOR PRECISE MEASUREMENT OF SURFACE WAVE VELOCITY WITH AN ACOUSTIC MICROSCOPE**

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New Techniques of digital signal processing have applied for  $V(z)$  curves to measure precisely leaky surface wave velocity,  $V_{lsw}$  with an acoustic microscope. The conventional approach to obtain  $V_{lsw}$  from a  $V(z)$  curve is Fast Fourier Transformation, in this approach, it is assumed that the ripple of  $V(z)$  curve extends infinitely. However, we observe only several oscillations in  $V(z)$  curves when we use an acoustic lens of 100-400MHz. Therefore it is preferable to choose other techniques of digital signal processing, which is applicable for finite numbers of oscillations.

The  $\Delta z$ , the period of the oscillations of  $V(z)$  curve, of fused silica and aluminum alloy has been calculated with the cross-correlation method, maximum entropy method, wavelet transformation. For the  $V(z)$  curve of four main oscillations of fused silica, the cross-correlation method gives a systematic increase in  $\Delta z$  for an decrease in the defocus distance  $z$ . This reflects the attenuation of acoustic and elastic waves with the change in the defocus distance. The maximum entropy method gives a sharper peak of  $\Delta z$ , thus it enables us to determine easily  $\Delta z$ . The wavelet transformation, which is applied for characterization of local similarity, depicts clearly the shift of the peak of  $\Delta z$  with an increase in the defocus distance  $z$ .

These new methods for calculating  $\Delta z$  make possible to measure precisely the  $V_{lsw}$  as well as the attenuation of the wave within very localized region near the surface.

### **ESTIMATION OF ULTRASONIC SOURCE DISTRIBUTIONS OF ELECTROACOUSTIC TRANSDUCERS**

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Analytical modelling of ultrasonic nondestructive evaluation (NDE) requires understanding of the propagation properties of ultrasonic fields [1, 2]. The aim of this

paper is to determine the source distribution of an electroacoustic transducer used to generate the considered ultrasonic fields. A modelling and identification approach is proposed to obtain the source distribution of an axisymmetric transducer from the measured data in the far field. The ultrasonic field produced by a transducer is modelled by the method of Gaussian beam superposition [3, 4]. The number and characteristic parameters of Gaussian beams for the superposition depend on the source distribution. An experimental setup is designed to scan the acoustic fields along a transverse plane to the propagation axis of the transducer using a small probe. In the setup, the spacing of sampling data points is chosen according to the sampling theorem so as to avoid aliasing distortion [5]. Taking into account the boundary conditions, a maximum likelihood estimator [6] is then utilized to determine the order of the model, i.e. the number of Gaussian beams, and to simultaneously estimate the coefficients of the field model from the measured amplitude and phase data. Once the order and coefficients of the field model are estimated, the source distribution of the transducer can easily be determined, and a complete description of the acoustic fields is then obtained. Using the estimated model, the prediction of the acoustic fields can be made in any transverse planes. The predicted and measured fields are then compared to validate the model. The method will be illustrated by both simulations and experiments.

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## **A NEW APPROACH TO ULTRASONIC IMAGE RECONSTRUCTION**

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Techniques of Quantitative Non-Destructive Evaluation (QNDE), which characterize a defect in size, shape and orientation, have been required in quality control of materials, in-service monitoring and estimation of residuary life expectancy of structures. The conventional pulse-echo method, which is the most popular method in ultrasonic NDE, has the lateral and depth resolutions that depend on the transducer size, the duration time of transmitted pulse and the transducer's position relative to a defect, so that usually it doesn't provide with enough resolution for the defect characterization.

Various ultrasonic techniques have been developed to improve the resolution and to characterize a defect. The Synthetic Aperture Focusing Technique (SAFT) and the Wiener filtering technique have been used for that purpose. Although improvement of a resolution, the SAFT result is still degraded by Point Spread Function (PSF). The Wiener filtering technique is one of deconvolution techniques that restore the degraded signal/image with using appropriate PSF. In ultrasonic NDE applications, however, any particular PSF of the raw B-scan data is valid over relatively narrow depth range because the ultrasonic beam spread varies with beam propagation, that is a defect depth.

A new approach for improving the resolution in ultrasonic NDE applications is presented in this paper. The newly implemented algorithm apply the Wiener filtering technique on a SAFT result to obtain a higher resolution. An advantage of the algorithm is not only to provide with a much higher resolution than the SAFT but also to require only one PSF, because a SAFT result has a depth invariant PSF. This paper describes the algorithm and subsequent results. The technique is compared with the SAFT and the Wiener filtering technique in the resolution and the signal-to-noise ratio. The effect of the resolution improvement is shown on simulated data numerically and experimental data.

## **WAVEFORM MAPPING OF PIEZOELECTRIC TRANSDUCER IMPULSE RESPONSES IN MULTI-TRANSDUCER PATTERN RECOGNITION-BASED UNDE SYSTEMS**

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This paper addresses a practical problem encountered with Ultrasonic Nondestructive Evaluation (UNDE) systems employing statistical or neural pattern recognition algorithms for material characterization or rapid component classification. The decision functions of a multi-transducer system, derived from empirical "training set" signals, depend on the impulse response of each piezoelectric transducer (PZT), as well as associated analog instrumentation. Signals acquired using through-transmission or

acousto-ultrasonic configurations are convolutions of the one-way impulse responses of the transmitting and receiving transducers, and the response of the material segment being nondestructively characterized. PZT impulse responses can change gradually with use, or abruptly, when malfunctioning transducers are replaced. Therefore, to avoid classification performance degradation, the affected decision algorithm must be "retrained" with the new transducer(s) re-applied to all of the original training set specimens.

An alternative method is presented which derives a compensatory linear filter, specific to each transducer/analog passband in the system. This waveform mapping derivation is only performed during system calibration, and requires measuring each two-way (pulse-echo) PZT response. The decision function then becomes independent of the individual PZT impulse responses, because the filters transform each into a "standard." Classification performance then becomes unaffected by PZT replacement or gradual aging.

The procedure requires that the "standard" two-way (pulse-echo) response is obtained by averaging time-aligned responses of all available transducers. During calibration, an iterative procedure, imposing time and frequency domain constraints, then deconvolves two-way pulse-echo data into one-way impulse responses. Wiener filters are derived for converting each one-way PZT response into the "standard." The iteration does not impact inspection through-put, because the transducer-specific Wiener filters performing the waveform mapping, can be combined with other computationally-efficient real time digital processing executed during on-line signal collection and material characterization.



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## Final Program Summary

### Monday, June 19

#### *Session (A) AM*

- I. **Magnetic Techniques**  
8:30-12:10

#### *Session (A) PM*

- II. **Non-Metallic Materials**  
1:30-4:50

#### *Session (B) PM*

- III. **Stress Measurement**  
8:30-12:10

#### *Session (B) PM*

- IV. **X-Ray Applications**  
1:30-4:30

### Tuesday, June 20

#### *Session (A) AM*

- V. **Optical Techniques Including  
Laser Ultrasound**  
8:30-12:10

#### *Session (B) AM*

- VI. **Thermal Techniques**  
8:30-12:10

#### **Posters**

1:30- 5:10

#### *Session (A) PM*

- VII. **Acoustic Emission and Internal  
Friction**  
1:30-4:50

### Wednesday, June 21

#### *Session (A) AM*

- VIII. **Acoustic Emission & Acousto-  
Ultrasonics**  
8:30-12:10

#### *Session (B) AM*

- IX. **Composite Materials**  
8:30-12:10

### Thursday, June 22

#### *Session (A) AM*

- X. **Process Control**  
8:30-12:10

#### *Session (A) PM*

- XI. **Particle Technology**  
1:30-4:30

#### *Session (B) AM*

- XII. **Ultrasonic Applications**  
8:30-12:10

#### *Session (B) PM*

- XIII. **Basic Ultrasonics**  
1:30-5:30